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SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

DIVISION DE NUEVAS CREACIONES

E.

S.

D.

Re: Expediente No. **08.053.594**

Solicitud de patente a nombre de **THE UNIVERSITY OF QUEENSLAND.**

Yo, DILIA MARIA RODRIGUEZ D'ALEMAN, identificada como aparece al pie de mi firma, domiciliada en la carrera 11 A No. 86-53, piso 6, obrando como apoderada de la sociedad **THE UNIVERSITY OF QUEENSLAND.**, domiciliada en Queensland, Australia, solicitante de la patente de la referencia, en respuesta al oficio No. **06988** notificado por fijación en lista el 4 de mayo de 2012, nos permitimos informar lo siguiente:

1. Con fundamento en la facultad que confiere el artículo 34 de la Decisión 486 al solicitante para modificar la solicitud de patente en cualquier momento del trámite, presentamos junto con este memorial nuevo pliego reivindicatorio que consta de 31 reivindicaciones dirigidas al método para determinar en un material geológico minerales.

En este sentido, el señor examinador podrá corroborar que se aclaró que la aplicación de la radiación microonda pulsada se hace con el detector de radiación térmico de acuerdo con lo estipulado en la memoria descriptiva.

Adicionalmente, se incluyeron las nuevas reivindicaciones 2, 3, 5, 7 y 25 con el fin de incluir las modalidades preferidas y para eliminar los términos "preferiblemente" del juego de reivindicaciones. El sustento para estas reivindicaciones se encuentra a lo largo de toda la memoria descriptiva y en las figuras 6 a 11 que muestran las áreas de mineralización sobre fragmentos de rocas.

Se eliminaron las antiguas reivindicaciones 9, 10 y 23.

Con relación al título de la invención, se acepta la amable sugerencia del señor examinador, luego el título de la invención queda así: “Método y Aparato para la determinación de la presencia de un mineral dentro de un material”.

2. CLARIDAD

En relación con las objeciones planteadas por el señor examinador, nos permitimos decir que con el nuevo capítulo reivindicatorio se superan todas las objeciones.

De otra parte, se revisó la traducción del último folio 43 y se comparó con el documento correspondiente en inglés (página 8, folio 11), sin embargo no se encontró inconsistencia entre las dos versiones. Agradecemos al señor examinador nos indique exactamente el aspecto que considera que requiere aclaración, ya que no parece haber errores involuntarios de traducción.

3. NOVEDAD

El señor examinador indica que la presente solicitud carece de novedad en vista de las enseñanzas de D1(US5170666); D2 (gb2206213) Y D3 (GB2188727).

En este sentido, me permito decir que el artículo 16 de la Decisión 486 de la Comisión de la Comunidad Andina establece que la invención se considera como nueva cuando no está comprendida en el estado de la técnica. Si de acuerdo con el espíritu de la norma legal indicada, la invención no se considera como nueva si está “comprendida” en el estado de la técnica, dicha norma se está refiriendo, naturalmente, a la totalidad del invento y no a alguno o a algunos de los componentes. Para que la invención pierda su calidad de novedosa, es claro que ella, TODA ELLA, ha de estar contenida en el “conjunto de conocimientos existentes que son aplicables al proceso industrial” como define el estado de la técnica el Tribunal de justicia del Acuerdo de Cartagena (Proceso No. 6-IP-89, sentencia de 31 de octubre de 1989, publicada en Jurisprudencia del Tribunal de Justicia del Acuerdo de Cartagena, Tomo 11, BID-INTAL, Buenos Aires, 1994, página 60).

Esto se conoce como NOVEDAD ABSOLUTA, de conformidad con lo cual para desvirtuar el requisito de novedad de una invención será necesario que lo reivindicado por la invención bajo estudio corresponda de forma total con lo revelado en un documento o anterioridad señalada, conclusión a la que se llega después de un análisis exhaustivo mediante una comparación entre el capítulo reivindicatorio de la invención en estudio y la divulgación de la referencia D1.

Ahora bien, si un determinado elemento, condición o circunstancia de la nueva creación ya hace parte del estado de la técnica no quiere ello decir, desde luego, que la invención deba ser rechazada por carecer del requisito de novedad. No sin razón ha dicho el mencionado Tribunal: “*hay que advertir que toda creación es en cierta forma, producto del estado de la técnica existente, en el sentido de que el inventor ha de tener en cuenta un conjunto de conocimientos técnicos que le permitan crear nuevos procedimientos aplicables a la industria*” (Ibídem, páginas 53 y 54) (el subrayado es nuestro).

Por lo tanto, como mínimo la novedad debe contemplar los siguientes aspectos:

-Que la invención en estudio esté contenida de forma **TOTAL y ABSOLUTA** en el documento que se menciona como anterioridad o de forma independiente en los documentos que se mencionan, si son varios.

-Que la invención en estudio puede partir de conocimientos conocidos, caso en el cual habrá que hacer la comparación no de lo conocido sino de lo inédito y novedoso, teniendo que analizarse el valor agregado que aporta la invención con sus nuevas características al campo técnico al cual pertenece la invención en estudio.

Aplicando lo anterior al caso concreto, para que las razones que aduce ese despacho para desvirtuar la novedad fuesen ciertas, sería forzoso que se demostrara que la invención de mi mandante esté comprendida en el estado de la técnica de forma **TOTAL**, lo cual no sucede con la materia reclamada en las nuevas reivindicaciones 1 a 30.

Se objetaron las reivindicaciones 1, 17 y 21 en vista de D1. Sobre este particular, nos permitimos decir que la reivindicación 1 y 21 son independientes en tanto que la cláusula anterior 17 depende de la reivindicación 1 ó la reivindicación 13 y ésta última no fue objetada.

No obstante lo anterior, nos permitimos decir que el documentos D1 no enseña o sugiere lo que el solicitante reclama en la presente invención.

En efecto, la nueva reivindicación 1 se encuentra dirigida a un método para la determinación de la presencia de un **mineral** en un **material geológico** que requiere entre otras cosas la detección de la radiación térmica emitida por el material **geológico** después de la aplicación de la radiación microonda pulsada por el detector de radiación térmico espaciado aparte del material geológico.

Por su parte, la reivindicación 21 ahora se encuentra dirigida al aparato para la determinación de la presencia de un mineral con un material geológico que requiere entre otras cosas un detector de radiación térmico espaciado aparte del material geológico para la detección de la radiación térmica emitida desde el material geológico.

Por su parte, D1 reporta la evaluación no-destructiva de materiales compuestos, y no materiales geológicos. El término materiales compuestos aparece en numerosos lugares en D1 que incluyen el resumen, el título de la invención, los antecedentes de la invención (columna 1, líneas 29-31), la descripción breve del arte previo (columna 1, líneas 48, 55, 61 y la columna 2, línea 4, resumen de la invención (columna 2, línea 23) y la descripción detallada (columna 3, líneas 15 a 16, columna 7, líneas 3-4, 36-37). Estos materiales compuestos incluyen esferas, varillas, placas, fibras de carbono-carbono, discos de freno, plásticos reforzados con vidrio, cerámicas etc (columna 7, líneas 3- 21).

En este sentido, el solicitante desea manifestar que los materiales son completamente diferentes de los materiales geológicos, como soporte el solicitante amablemente dirige la atención del señor examinador a un extracto del documento de Chung 2010 (ANEXO 1) el cual indica que:

“Composite materials are multiphase materials obtained through the artificial combination of different materials in order to attain properties that the individual components by themselves cannot attain. They are not multiphase materials in which the different phases are formed naturally by reactions, phase transformations, or other phenomena.” (véase página 1, el subrayado es nuestro).

También el señor examinador está refiriéndose al extracto adjunto de Hunt 2007 (ANEXO 2) el cual indica que se definen dos constituyentes generales de materiales geológicos, es decir, rocas y suelos como. “[m]aterial of the Earth’s crust, composed of one or more minerals strongly bonded together”. Soil is “a naturally occurring mass of discrete particles or grains, at most lightly bonded together, occurring as a product of rock weathering” (página 3). Esta definición de roca es consistente con la definición proporcionada con la Enciclopedia Británica:

“Rock, in geology, naturally occurring and coherent aggregate of one or more minerals. Such aggregates constitute the basic units of which the solid Earth is comprised and typically form recognizable and mappable volumes.”

Respecto a las nuevas reivindicaciones 3 y 27, es necesario señalar que la Enciclopedia Británica también define mineral como “an aggregate of economically

important minerals sufficiently rich to enable separation of the minerals at a profit'. Se adjunta (ANEXO 3) una copia del extracto de la Enciclopedia Britanica.

En vista de estas definiciones de materiales geológicos, rocas, suelo y mineral, nos permitimos decir que una persona medianamente versada en la materia técnica que está familiarizada con dichos términos entendería que su estructura, propiedades físicas y químicas son muy diferentes de los materiales compuestos enunciado en D1.

Como se indicó más arriba, D1 reporta la evaluación no destructiva de materiales compuestos. Un material compuesto no puede ser considerado o equiparado con un material geológico y por lo tanto, D1 falla en enseñar o sugerir la evaluación no destructiva de **materiales geológicos**.

Aunado a lo anterior, también es necesario señalar que las reivindicaciones independientes 1 y 21 también requieren la determinación de la presencia de un **mineral** en un material geológico. La presente memoria descriptiva proporciona a la persona versada en la materia técnica la guía necesaria en cuanto al significado y alcance del término "mineral", en contraste con D1 que falla en enseñar o sugerir la determinación de la presencia de un mineral.

Aún otra diferencia entre la reivindicación 1 y 21 con lo expuesto en D1 es que las reivindicaciones 1 y 21 requieren la detección de la radiación térmica emitida desde el material geológico mediante el detector de radiación térmica espaciado aparte del material geológico. D1 falla en enseñar o sugerir este requerimiento. Más aún, D1 enseña que la detección de **ondas elásticas** que se propagan sobre la superficie de un espécimen es por medio de un detector que es **físicamente conectado** con la superficie del espécimen (véase por favor la columna 2, líneas 20 a 34, columna 3, líneas 19-40 y figuras 1, 3 y 4). En este sentido, es claro que las ondas elásticas no pueden ser equiparables a la radiación térmica.

Por todas las razones anteriormente expuestas, es claro que D1 falla en enseñar o sugerir el método y el aparato de las reivindicaciones independientes 1 y 21 de la presente solicitud. Más aún, D1 se aleja del método de la reivindicación 1 porque enseña un método para la evaluación no destructiva de un espécimen de **material compuesto** que involucra la transmisión de microondas pulsada hacia el espécimen para producir una **onda elástica** que se propaga sobre la superficie del espécimen. La onda elástica es detectada por un detector **físicamente conectado** a la superficie del espécimen. La conexión entre el detector y la superficie del espécimen es crítica para el método de la anterioridad D1.

Sobre este particular, es necesario señalar que si el detector no está conectado con la superficie del espécimen de material compuesto, la onda elástica no será detectada y no será posible la evaluación. Se genera la correspondiente señal y se analiza a fin de clasificar el espécimen con referencia a las muestras defectivas y no defectivas (véase por favor la columna 2, líneas 20-34, columna 3, líneas 19-40, figuras 1, 3 y 4 de D1).

En este sentido, es claro que la persona medianamente versada en la técnica que tuviera acceso a D1 no encontraría evidente modificar el método de la anterior D1 para llegar de manera evidente a la materia definida por la presente invención. De hecho, D1 no proporcionaría ninguna motivación a dicha persona para alterar el método allí reportado para sustituir el material compuesto por material geológico, o la detección de onda elástica por detección de radiación térmica y la localización alejada del detector de la superficie del espécimen.

Ahora, en relación con los documentos D2 y D3, nos permitimos decir que estas dos anterioridades reportan un método y un aparato para clasificación de materiales minerales que involucra la irradiación de una partícula de mineral con radiación microonda y la medición de la radiación emitida desde la partícula de mineral por medio de un detector (51 en la figura de D2 y 20 en la figura de D3) localizado fijamente sobre la partícula de mineral.

No obstante, D2 y D3 fallan en enseñar o sugerir un método y un aparato que involucre la radiación microonda **pulsada** como se requiere en todas las reivindicaciones de la presente solicitud. Como se explica a lo largo de la memoria descriptiva de la presente solicitud y en las figuras 2 a 11, el solicitante ha encontrado de manera sorprendente que el uso de radiación microonda **pulsada** en la presente invención proporciona resultados superiores que usando irradiación microonda constante. Tal ventaja inesperada no habría podido ser prevista o anticipada a partir de lo enseñado en D2 y D3.

Sobre este particular, también se hace necesario hacer énfasis sobre el hecho que D2 falla en enseñar o incluso sugerir un **detector de radiación térmico** espaciado aparte del material geológico. Por el contrario, D2 lo que reporta es una antena de guía de onda 51 para **detectar las microondas** transmitidas a través de las partículas. D2 no reporta la detección de radiación térmica como si lo hace la presente invención en sus reivindicaciones independientes 1 y 21.

Así las cosas, la presente invención cumple a cabalidad con los requisitos de patentabilidad indicados por la Decisión 486 de la comunidad Andina.

Finalmente, nos permitimos manifestar que se conceda finalmente el privilegio de patente de invención a la solicitud en estudio o si el examinador considera que subsisten razones para objetar la presente invención, rogamos expedir otro examen de fondo con los debidos motivos y razones que sustenten su objeción con el fin de que nuestro mandante tenga la posibilidad de exponer sus argumentos con base en la interpretación de los artículos 45 de la Decisión 486 -el cual otorga la facultad al examinador para emitir los exámenes de fondo-, 35 y 59 del Código Contencioso Administrativo que señala que: *"habiéndose dado oportunidad a los interesados para expresar sus opiniones, y con base en las pruebas e informes disponibles, se tomará la decisión que será motivada al menos en forma sumaria si afecta a particulares"* y *"(.) deberá proferirse la decisión definitiva. Esta se motivará en sus aspectos de hecho y de derecho, y en los de conveniencia si es el caso"*, respectivamente (El subrayado es nuestro).

En consecuencia, pedimos a ese despacho pronunciarse en relación con todos los argumentos presentados en este memorial dando estricta aplicación al principio del debido proceso consagrado en el artículo 29 de la Constitución Nacional.

Señor Superintendente,



DILIA MARIA RODRIGUEZ D'ALEMAN
C.C. 41.654.882 de Bogotá
T.P. 20824 del C.S.J.

REIVINDICACIONES

1. Un método para determinar la presencia de un mineral en un material geológico CARACTERIZADO PORQUE incluye:
Aplicar radiación microonda pulsada al material geológico;
Detectar radiación térmica emitida desde el material geológico después de la aplicación de la radiación microonda pulsada por un detector de radiación térmico espaciado aparte del material geológico; y
Analizar la radiación térmica detectada para determinar la presencia del mineral en el material geológico.
2. Un método de acuerdo con la reivindicación 1, caracterizado porque el paso de análisis de la radiación térmica detectada incluye determinar las áreas de mineralización en el material geológico.
3. Un método de acuerdo con la reivindicación 1, caracterizado porque el material geológico incluye rocas, fragmentos de roca, mineral o fragmentos de mineral.
4. Un método de acuerdo con la reivindicación 1, caracterizado porque la radiación microonda pulsada aplicada al material geológico incluye un pulso de hasta 1 segundo de duración.
5. Un método de acuerdo con la reivindicación 1, caracterizado porque la radiación microonda pulsada aplicada al material geológico incluye un pulso de desde 0.1 hasta 1 segundo de duración.
6. Un método de acuerdo con la reivindicación 1, caracterizado porque la radiación microonda pulsada aplicada al material geológico incluye una pluralidad de pulsos de hasta 1 segundo de duración.
7. Un método de acuerdo con la reivindicación 1, caracterizado porque la radiación microonda pulsada aplicada al material geológico incluye una pluralidad de pulsos de desde 0.1 hasta 1 segundo de duración.

8. Un método de acuerdo con la reivindicación 1, caracterizado porque la radiación microonda pulsada tiene una densidad de potencia inducida dentro de los minerales objetivos por debajo de 1000 MW/m^3 .
9. Un método de acuerdo con la reivindicación 8, caracterizado porque la radiación microonda pulsada tiene una densidad de potencia inducida dentro de los minerales objetivo desde 1 hasta 100 MW/m^3 .
10. Un método de acuerdo con la reivindicación 1 caracterizado porque la radiación microonda pulsada tiene una frecuencia que varía desde 900 hasta 5800 MHz.
11. Un método de acuerdo con la reivindicación 10, caracterizado porque la radiación microonda pulsada tiene una frecuencia que varía desde 900 hasta 3500 MHz.
12. Un método de acuerdo con la reivindicación 11, caracterizado porque la radiación microonda pulsada tiene una frecuencia que varía desde 915 hasta 950 MHz.
13. Un método de acuerdo con la reivindicación 1 caracterizado porque el material geológico es alimentado hacia una zona de irradiación microonda para su posterior aplicación de radiación microonda pulsada.
14. Un método de acuerdo con la reivindicación 13, caracterizado porque el material geológico es alimentado hacia la zona de irradiación microonda en un canal de alimentación.
15. Un método para determinar la presencia de un mineral en un material geológico caracterizado porque incluye:
Aplicar radiación microonda pulsada de una primera frecuencia al material;
Detectar una primera cantidad de radiación emitida desde el material geológico después de la aplicación de la radiación microonda pulsada de la primera frecuencia;

Aplicar radiación microonda pulsada de una segunda frecuencia al material;

Detectar una segunda cantidad de radiación emitida desde el material después de la aplicación de la radiación microonda pulsada de la segunda frecuencia; y

Analizar la primera y segunda cantidad detectada de radiación, a fin de determinar la presencia del mineral dentro del material.

16.Un método de acuerdo con la reivindicación 15, caracterizado porque la primera frecuencia varía desde 920 hasta 925 y la segunda frecuencia varía desde 915 hasta 920 MHZ.

17.Un método de acuerdo con la reivindicación 15 caracterizado porque el análisis de la primera y segunda cantidad de radiación incluye una substracción de una de las cantidades de radiación de la otra.

18.Un método de acuerdo con la reivindicación 15, caracterizado porque incluye una aplicación adicional de radiación microonda de una frecuencia diferente a la primera y segunda frecuencia al material y una detección posterior de la cantidad de radiación emitida desde el material.

19.Un método para clasificar un flujo de alimentación de material hacia una pluralidad de flujos, caracterizado porque incluye:

Determinar la presencia de un mineral en el flujo de alimentación del material usando un método de acuerdo con la reivindicación 1 o la reivindicación 15; y

Separar el flujo de alimentación de material al menos hacia la pluralidad de flujos basados en esa determinación.

20.Un método de acuerdo con la reivindicación 19, caracterizado porque el flujo de alimentación se separa en un flujo de desperdicio y un flujo de producto.

21. Un método de acuerdo con la reivindicación 19 caracterizado porque el flujo de alimentación se separa en un flujo de desperdicio y una pluralidad de flujos de producto basado en el grado mineral del material.
22. Un método según la reivindicación 19 caracterizado porque el flujo de alimentación se separa en una pluralidad de flujos mediante un compresor de aire de alta presión.
23. Un aparato para determinar la presencia de un mineral dentro de un material geológico, caracterizado porque incluye:
- Una zona de irradiación microonda que incluye un emisor de microonda adaptado para emitir radiación microonda pulsada hacia la zona de irradiación microonda;
- Un detector de radiación térmico espaciado aparte del material geológico para detectar la radiación térmica emitida desde el material geológico que sigue la aplicación de radiación microonda pulsada al material geológico; y
- Un analizador para interpretar la radiación térmica detectada y por lo tanto determinar la presencia del mineral dentro del material geológico.
24. Un aparato de acuerdo con la reivindicación 23 caracterizado porque el detector de radiación térmico es capaz de determinar las áreas de mineralización en el material geológico.
25. Un aparato según la reivindicación 23, caracterizado porque el emisor de microondas es una cavidad microonda en modo individual o modo múltiple.
26. Un aparato según la reivindicación 23, caracterizado porque el analizador es un computador equipado con un software que procesa la imagen.
27. Un aparato según la reivindicación 23 caracterizado porque el material geológico incluye rocas o fragmentos de roca, mineral o fragmentos de

mineral y el aparato incluye una cámara visible a la luz para medir el tamaño de las rocas, los fragmentos de roca, los minerales o los fragmentos de minerales.

28.Un aparato según la reivindicación 23, caracterizado porque incluye un clasificador para seleccionar el material geológico hacia la pluralidad de flujos.

29.Un aparato según la reivindicación 28 caracterizado porque el clasificador es un compresor de aire de alta presión.

30.Un aparato según la reivindicación 23, caracterizado porque incluye un alimentador para suministrar el material geológico a la zona de irradiación microonda en un canal.

31.Un aparato según la reivindicación 30, caracterizado porque el alimentador es una correa transportadora.

ANEXO 1

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University at Buffalo,
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With thanksgiving for the past twenty years and excitement for the future, I dedicate this book to the Composite Materials Research Laboratory, which I founded at the University at Buffalo, State University of New York, in 1989. With the highly appreciated involvement of a large number of researchers, including students, postdoctoral associates, visiting scholars, faculty members and industrial participants, the Laboratory has made advances in the science and technology of composite materials, as shown by 400 peer-reviewed journal publications that cover composites with polymer, cement, and metal matrices, including those for aerospace, automotive, civil, electronic, energy and thermal applications. The Laboratory has emphasized material development that is application driven and process oriented, as partly described in this book. Our most notable breakthroughs relate to smart concrete, vibration damping materials, cement-based pn-junctions, thermoelectric materials, multifunctional structural materials, carbon nanofiber applications, electromagnetic interference shielding materials, electronic packaging materials and thermal interface materials. The graduation of over 30 doctoral students is particularly heartwarming. Special appreciation is extended to Mr. Mark A. Lukowski, for twenty years of technical support.

In further celebration of the anniversary, the Laboratory has commissioned Kenneth C.K. Yip (composer) and I (lyrics and narration writer) to write a musical that merges science and music. The piece is entitled *Materials Are Needed to Make Anything*, and is for performance by a chorus and two vocal soloists and percussion accompaniment. This is the first musical work that centers on engineering materials! Choirs interested in performing this 30-minute piece are welcome to contact me.

Deborah D.L. Chung
Director and Professor
Composite Materials Research Laboratory
Buffalo, NY
June 1, 2009

Preface to the Second Edition

The field of composite materials has progressed greatly over the last few decades, as shown by the widespread use of fibrous composite materials for airframes, sporting goods and other lightweight structures. Enabling this technological progress is scientific understanding of the design and mechanics of composite materials that involve continuous fibers as the reinforcement.

Current challenges in the field of composite materials are associated with the extension of the field of composite materials from structural composites to functional and multifunctional composites, the development of composite materials for electrical, thermal and other functional applications that are relevant to current technological needs, and the improvement of composite materials through processing. Examples of functions are joining (e.g., brazing), repair, sensing, actuation, deicing (as needed for aircraft and bridges), energy conversion (as needed to generate clean energy), electrochemical electrodes, electrical connection, thermal contact improvement and heat dissipation (i.e., cooling, as needed for microelectronics and aircraft). Processing includes the use of additives (which may be introduced as liquids or solids), the combined use of fillers (including discontinuous ones) at the micrometer and nanometer scales, the formation of hybrids (such as organic–inorganic hybrids), the modification of the interfaces in a composite, and control over the microstructure. In other words, the development of composite materials for current technological needs must be application driven and process oriented. This is in contrast to the conventional composites engineering approach, which focuses on mechanics and purely structural applications.

The second edition is aimed at providing in an organized manner the concepts and technical details for addressing the challenges mentioned above. Compared to the first edition, the second edition has been greatly expanded (with hundreds of illustrations added), extensively reorganized and almost totally rewritten.

As a part of a comprehensive treatment of composite tailoring methods, numerous examples of methods, in addition to data and micrographs that support their effectiveness, have been added to the second

edition. Topics added to the second edition include vibration damping, degradation, durability, mechanical joining, electrical connection and nanocomposites. Composites with polymer, cement, metal and carbon matrices are covered in terms of the structure, properties, fabrication and applications. Up-to-date information on lightweight structural materials and civil infrastructure materials is provided. Materials for structural, electrical and thermal applications are comprehensively covered with much information that is not in the first edition. The emphasis on process-oriented composite design, application-oriented functional properties and multifunctional structural materials makes this book different from all other books on composite materials.

Due to the large amount of up-to-date and down-to-earth information, the book is also suitable for use as a reference book for students and professionals that are interested in the development of composite materials. Due to the tutorial style, basic concept coverage, example problems and review questions, the book is also suitable for use as a textbook for undergraduate and graduate students that have had a semester of an introductory materials science course. The relevant academic disciplines include materials, chemical, mechanical, aerospace, electrical and civil engineering.

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April 16, 2009

<http://alum.mit.edu/www/ddlchung>

Preface to the First Edition

Books on composite materials are essentially limited to addressing the fabrication and mechanical properties of these materials, because of the dominance of structural applications (such as aerospace applications) for traditional composite materials. However, nonstructural applications are rapidly increasing in importance due to the needs of the electronic, thermal, battery, biomedical and other industries. The scientific concepts that guide the design of functional composites and that of structural composites are quite different, as both the performance and cost requirements are different. Therefore, this book is different from related books in its emphasis on functional composite materials. The functions addressed include structural, thermal, electrical, electromagnetic, thermoelectric, electromechanical, dielectric, magnetic, optical, electrochemical and biomedical functions. The book provides the fundamental concepts behind the ability to provide each function, in addition to covering the fabrication, structure, properties and applications of the relevant composite materials.

Books on composite materials tend to emphasize polymer-matrix composites, though cement-matrix composites are the most widely used structural materials, and metal-matrix, carbon-matrix and ceramic-matrix composites are rising in importance. In contrast, this book covers composite materials with all of the abovementioned matrices.

The most common approach for books on composite materials is to emphasize mechanics issues, due to the relevance of mechanics to structural applications. A less common approach for books on composite materials is to categorize composite materials in terms of their matrices and cover the composites in accordance with their matrix materials. In contrast, this book takes a new approach, namely the functional approach—covering composites in accordance with their functions. The functional approach allows the readers to appreciate how composites are designed for the needs of various industries. Such appreciation is valuable for students who are preparing themselves for industrial positions (most students are) and for professionals working in various industries. Moreover, the functional approach allows

an organized presentation of numerous scientific concepts other than those related to mechanical behavior, thereby enabling a wide scientific scope to be covered.

The book is tutorial in style, but it is up-to-date, and each chapter includes an extensive list of references. The readers need to have taken a course on introductory materials science. They do not need to have taken any prior course on composite materials. Therefore, the book is suitable for use as a textbook for upper-level undergraduate students and for graduate students. It is also suitable for use as a reference book for students, engineers, technicians, technology managers and marketing personnel.

Because of the wide scientific scope enabled by the functional approach, the book is expected to be useful to all kinds of engineers (including electrical, thermal, chemical and industrial engineers). In contrast, the conventional approach that emphasizes mechanical behavior limits the readership to materials, mechanical, aerospace and civil engineers.

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April 1, 2002

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- Corrosion resistance
- Dimensional stability (can be designed for zero CTE)
- Vibration damping ability
- Low electrical resistivity
- High electromagnetic interference (EMI) shielding effectiveness
- High thermal conductivity.

Another example of a composite is concrete, which is a structural composite obtained by combining (through mixing) cement (the matrix, i.e., the binder, obtained by a reaction – known as hydration – between cement and water), sand (fine aggregate), gravel (coarse aggregate), and optionally other ingredients that are known as admixtures. Short fibers and silica fume (a fine SiO_2 particulate) are examples of admixtures. In general, composites are classified according to their matrix material. The main classes of composites are polymer-matrix, cement-matrix, metal-matrix, carbon-matrix and ceramic-matrix composites.

Polymer-matrix and cement-matrix composites are the most common, due to the low cost of fabrication. Polymer-matrix composites are used for lightweight structures (aircraft, sporting goods, wheel chairs, etc.), in addition to vibration damping, electronic enclosures, asphalt (composite with pitch, a polymer, as the matrix), solder replacement, etc. Cement-matrix composites in the form of concrete (with fine and coarse aggregates), steel-reinforced concrete, mortar (with fine aggregate, but no coarse aggregate) or cement paste (without any aggregate) are used for civil structures, prefabricated housing, architectural precasts, masonry, landfill cover, thermal insulation, sound absorption, etc.

Carbon-matrix composites are important for lightweight structures (e.g., Space Shuttles) and components (e.g., aircraft brakes) that need to withstand high temperatures, but they are relatively expensive due to the high cost of fabrication. Carbon-matrix composites suffer from their tendency to be oxidized ($2\text{C} + \text{O}_2 \rightarrow 2\text{CO}$), thereby becoming vapor.

Carbon fiber carbon-matrix composites, also called carbon-carbon composites, are the most advanced form of carbon, as the carbon fiber reinforcement makes them stronger, tougher, and more resistant to thermal shock than conventional graphite. With the low density of carbon, the specific strength (strength/density), specific modulus (modulus/density) and specific thermal conductivity (thermal conductivity/density) of carbon-carbon composites are the highest among composites. Furthermore, the coefficient of thermal expansion is near zero.

Ceramic-matrix composites are superior to carbon-matrix composites in terms of oxidation resistance, but they are not as well developed as carbon-matrix composites. Metal-matrix composites with aluminum as the matrix are used for lightweight structures and low-thermal-expansion electronic enclosures, but their applications are limited by the high cost of fabrication and by galvanic corrosion.

Metal-matrix composites are gaining importance because the reinforcement serves to reduce the coefficient of thermal expansion (CTE) and increase the strength and modulus. If a relatively graphitic kind of carbon fiber is used, the thermal conductivity can also be enhanced. The combination of low CTE and high

1 Composite Material Structure and Processing

1.1 Introduction

Composite materials are multiphase materials obtained through the artificial combination of different materials in order to attain properties that the individual components by themselves cannot attain. They are not multiphase materials in which the different phases are formed naturally by reactions, phase transformations, or other phenomena. An example is carbon fiber reinforced polymer. Composite materials should be distinguished from alloys, which can comprise two more components but are formed naturally through processes such as casting. Composite materials can be tailored for various properties by appropriately choosing their components, their proportions, their distributions, their morphologies, their degrees of crystallinity, their crystallographic textures, as well as the structure and composition of the interface between components. Due to this strong tailorability, composite materials can be designed to satisfy the needs of technologies relating to the aerospace, automobile, electronics, construction, energy, biomedical and other industries. As a result, composite materials constitute most commercial engineering materials.

An example of a composite material is a lightweight structural composite that is obtained by embedding continuous carbon fibers in one or more orientations in a polymer matrix. The fibers provide the strength and stiffness, while the polymer serves as the binder. In particular, carbon fiber polymer-matrix composites have the following attractive properties:

- Low density (lower than aluminum)
- High strength (as strong as high-strength steels)
- High stiffness (stiffer than titanium, yet much lower in density)
- Good fatigue resistance
- Good creep resistance
- Low friction coefficient and good wear resistance
- Toughness and damage tolerance (as enabled by using appropriate fiber orientations)
- Chemical resistance (chemical resistance controlled by the polymer matrix)

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thermal conductivity makes them very attractive for electronic packaging (e.g., heat sinks). Besides good thermal properties, their low density makes them particularly desirable for aerospace electronics and orbiting space structures; orbiters are thermally cycled by moving through the Earth's shadow.

Compared to the metal itself, a carbon fiber metal-matrix composite is characterized by a higher strength-to-density ratio (i.e., specific strength), a higher modulus-to-density ratio (i.e., specific modulus), better fatigue resistance, better high-temperature mechanical properties (a higher strength and a lower creep rate), a lower CTE, and better wear resistance.

Compared to carbon fiber polymer-matrix composites, a carbon fiber metal-matrix composite is characterized by higher temperature resistance, higher fire resistance, higher transverse strength and modulus, a lack of moisture absorption, a higher thermal conductivity, a lower electrical resistivity, better radiation resistance, and absence of outgassing.

On the other hand, a metal-matrix composite has the following disadvantages compared to the metal itself and the corresponding polymer-matrix composite: higher fabrication cost and limited service experience.

Fibers used for load-bearing metal-matrix composites are mostly in the form of continuous fibers, but short fibers are also used. The matrices used include aluminum, magnesium, copper, nickel, tin alloys, silver-copper, and lead alloys. Aluminum is by far the most widely used matrix metal because of its low density, low melting temperature (which makes composite fabrication and joining relatively convenient), low cost, and good machinability. Magnesium is comparably low in melting temperature, but its density is even lower than aluminum. Applications include structures (aluminum, magnesium), electronic heat sinks and substrates (aluminum, copper), soldering and bearings (tin alloys), brazing (silver-copper), and high-temperature applications (nickel).

Although cement is a ceramic material, ceramic-matrix composites usually refer to those with silicon carbide, silicon nitride, alumina, mullite, glasses and other ceramic matrices that are not cement.

Ceramic-matrix fiber composites are gaining increasing attention because the good oxidation resistance of the ceramic matrix (compared to a carbon matrix) makes the composites attractive for high-temperature applications (e.g., aerospace and engine components). The fibers serve mainly to increase the toughness and strength (tensile and flexural) of the composite due to their tendency to be partially pulled out during the deformation. This pullout absorbs energy, thereby toughening the composite. Although the fiber pullout is advantageous, the bonding between the fibers and the matrix must still be sufficiently strong for the fibers to strengthen the composite effectively. Therefore, control over the bonding between the fibers and the matrix is important for the development of these composites.

When the reinforcement is provided by carbon fibers, the reinforcement has a second function, which is to increase the thermal conductivity of the composite, as the ceramic is mostly thermally insulating whereas carbon fibers are thermally conductive. In electronic, aerospace, and engine components, the enhanced thermal conductivity is attractive for heat dissipation.

A third function of the reinforcement is to decrease the drying shrinkage in the case of ceramic matrices prepared using slurries or slips. In general, the drying shrinkage decreases with increasing solid content in the slurry. Fibers are more effective than particles at decreasing the drying shrinkage. This function is attractive for the dimensional control of parts made from the composites.

Fiber-reinforced glasses are useful for space structural applications, such as mirror back structures and supports, booms and antenna structures. In low Earth orbit, these structures experience a temperature range from -100 to 80°C , so they need an improved thermal conductivity and a reduced coefficient of thermal expansion. In addition, increased toughness, strength and modulus are desirable. Due to the environmental degradation resistance of carbon fiber reinforced glasses, they are also potentially useful for gas turbine engine components. Additional attractions are low friction, high wear resistance, and low density.

The glass matrices used for fiber-reinforced glasses include borosilicate glasses (e.g., Pyrex), aluminosilicate glasses, soda lime glasses and fused quartz. Moreover, a lithia aluminosilicate glass-ceramic and a $\text{CaO-MgO-Al}_2\text{O}_3\text{-SiO}_2$ glass-ceramic have been used.

1.2 Composite Material Structure

The structure of a composite is commonly such that one of the components is the matrix while the other components are fillers bound by the matrix, which is often called the binder. For example, in carbon fiber reinforced polymer, which is important for lightweight structures, the polymer is the matrix, while the carbon fiber is the filler. In case of a structural composite, the filler usually serves as a reinforcement. For example, carbon fiber is a reinforcement in the polymer-matrix composite.

Composites can be classified according to the matrix material, which can be a polymer, a metal, a carbon, a ceramic or a cement (e.g., Portland cement). They can also be classified according to the shape of the filler. A composite that has particles as the filler is said to be a particulate composite. For example, concrete is a particulate composite in which cement is the matrix and sand and stones are the two types of particles that are present together. A composite with fibers used as the filler is said to be a fibrous composite.

The components in a composite can also take the form of layers. An example is laminate flooring that consists of layers of polymer, paper and fiberboard that are joined together during fabrication.

A composite material can be in bulk or film form. The film form can be such that the composite is a standalone film or a film that is attached to a substrate. Less commonly, a composite material takes the form of particles or fibers; i.e., a single particle or fiber consisting of more than one component.

1.2.1 Continuous Fiber Composites

A fibrous composite involving continuous fibers is particularly attractive as a structural material due to the high strength and modulus of the fibers, which bear most of the load. A continuous carbon fiber polymer-matrix composite is an example. The use of steel reinforcing bars (called “rebars”) to reinforce concrete gives steel reinforced concrete, which is another example (even though steel rebars are not referred to as fibers). A fibrous composite is also attractive in that it can be tailored by choosing the orientation of the fibers. A common configuration involves the fibers being in the form of plies. A ply, also known as a lamina, is a sheet that has fibers with the same orientation in the plane of the sheet. Each lamina has thousands of fibers along its thickness because each fiber tow consists of thousands of fibers. The composite is made up of a number of laminae such that the fiber orientations can be different among the laminae. For example, the fibers in the consecutive laminae can be oriented at 0, 90, +45 and -45°, resulting in a two-dimensionally “quasi-isotropic” configuration. A conventional system of notation for describing the lay-up configuration of the laminae is illustrated below.

[0]₈ means an eight-lamina composite with all laminae having fibers in the same direction (0°). [0/90]_{2s} (where the subscript “s” means “symmetric”) means an eight-lamina composite with the stacking order 0, 90, 0, 90, 90, 0, 90, 0°, where the first four laminae and the remaining four laminae are mirror images and the mirror plane is the center plane of the composite. [0/45/90/-45]_s means an eight-lamina composite with the stacking order 0, 45, 90, -45, -45, 90, 45, 0°, where the first four laminae and the remaining four laminae are mirror images. [0/45/90/-45]_{2s} means a 16-lamina composite with the stacking order 0, 45, 90, -45, 0, 45, 90, -45, -45, 90, 45, 0, -45, 90, 45, 0°, where the first eight laminae and the remaining eight laminae are mirror images. [0/45/90/-45]_{2s} means a 16-lamina composite with the stacking order 0, 45, 90, -45, 0, 45, 90, -45, -45, 90, 45, 0, -45, 90, 45, 0°, where the first eight laminae and the remaining eight laminae are mirror images. [0/45/90/-45]_{3s} means a 24-lamina composite with the stacking order 0, 45, 90, -45, 0, 45, 90, -45, 0, 45, 90, -45, -45, 90, 45, 0, -45, 90, 45, 0, -45, 90, 45, 0, -45, 90, 45, 0°, where the first 12 laminae and the remaining 12 laminae are mirror images.

An optical micrograph of the interlaminar interface between two laminae that are at 90° to one another (i.e., a crossply configuration). The interlaminar interface is the region between the two parallel lines that are separated by 8.7 μm. The fibers above the interface are in the plane of the paper, whereas those below the interface are perpendicular to the paper.

The direction perpendicular to the laminae is known as the through-thickness direction. The interface between two adjacent laminae is known as the interlaminar interface, which is the mechanically weak link in the laminate. Figure 1.1 is an optical micrograph of the interlaminar interface between two laminae that are 90° relative to one another (i.e., a crossply configuration). This means that the through-thickness direction is relatively weak mechanically, and delamination (local separation of the laminae from one another) is a common form of damage in these composites. When the fibers are carbon fibers, which are much more conductive electrically than the polymer matrix, the through-thickness direction

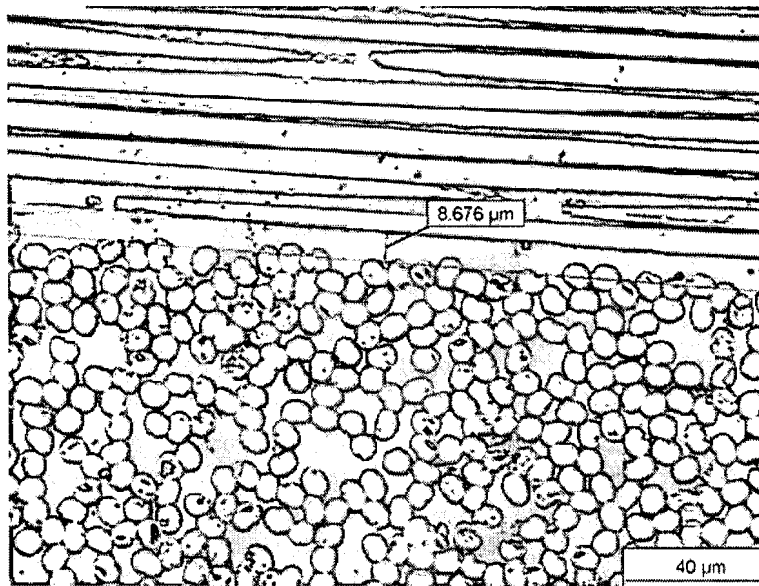


Figure 1.1. An optical micrograph of the interlaminar interface between two laminae that are at 90° to one another (i.e., a crossply configuration). The interlaminar interface is the region between the two parallel lines that are separated by $8.7\text{ }\mu\text{m}$. The fibers above the interface are in the plane of the paper, whereas those below the interface are perpendicular to the paper

also has a relatively high electrical resistivity (i.e., low electrical conductivity). In other words, the composites are strongly anisotropic (i.e., the properties are different in different directions) both mechanically and electrically.

1.2.2 Carbon–Carbon Composites

The carbon fibers used for carbon–carbon composites are usually continuous and woven. Both two-dimensional and higher-dimensional weaves are used, though the latter has the advantage of enhanced interlaminar shear strength.

The weave pattern of the carbon fabric affects the densification of the carbon–carbon composite during composite fabrication. An 8H satin weave is preferred over a plain weave because of the inhomogeneous matrix distribution around the crossed bundles in the plain weave. Microcracks tend to develop beneath the bundle crossover points.

For two-dimensional carbon–carbon composites containing plain weave fabric reinforcements under tension, the mode of failure of the fiber bundles depends on their curvature. Fiber bundles with small curvatures fail due to tensile stress or due to a combination of tensile and bending stresses. Fiber bundles with large curvatures fail due to shear stresses at the point where the local fiber direction is most inclined to the applied load.

Circular fibers are preferred to irregularly shaped fibers, as the latter leads to stress concentration points in the matrix around the fiber corners. Microcrack initiation occurs at these points, thus resulting in low strength in the carbon–carbon composite.

1.2.3 Cement-Matrix Composites

Cement-matrix composites include concrete, which is a cement-matrix composite with a fine aggregate (sand), a coarse aggregate (gravel) and optionally other additives (called admixtures). Concrete is the most widely used civil structural material. When the coarse aggregate is absent, the composite is known as a mortar, which is used in masonry (for joining bricks) and for filling cracks. When both coarse and fine aggregates are absent, the material is known as cement paste. Cement paste is rigid after curing (the hydration reaction involving cement – a silicate – and water to form a rigid gel).

The admixtures can be a fine particulate such as silica (SiO_2) fume to decrease the porosity in the composite. It can be a polymer (used in either a liquid solution form or a solid dispersion form) such as latex, again to decrease the porosity. It can be short fibers (such as carbon fibers, glass fibers, polymer fibers and steel fibers) to increase the toughness and decrease the drying shrinkage (shrinkage during curing – undesirable, as it can cause cracks to form). Continuous fibers are seldom used because of their high cost and the impossibility of incorporating continuous fibers into a cement mix. Due to the bidding system used for many construction projects, low cost is essential.

Fibrous cement-matrix composites are structural materials that are gaining in importance quite rapidly due to the increasing demand for superior structural and functional properties. Discontinuous fibers used in concrete include steel, glass, polymer and carbon fibers. Among these fibers, carbon and glass fibers are micrometer scale (e.g., $10\mu\text{m}$) in diameter, whereas steel and polymer fibers are usually much larger in diameter (e.g., $100\mu\text{m}$). For the microfibers, the fiber length is typically around 5 mm, as fiber dispersion becomes more difficult as the fiber length increases. Due to the weak bond between fiber and the cement matrix, continuous fibers are much more effective than short fibers at reinforcing concrete. However, continuous fibers cannot be incorporated into a concrete mix, and it is difficult for the concrete mix to penetrate into the space between adjacent fibers, even in the absence of aggregates. The alignment of the continuous fibers in concrete also adds to the implementation difficulty. Therefore, short fibers are typically used.

The effect of short fiber addition on the properties of cement increases with increasing fiber volume fraction unless the fiber volume fraction is so high that the air void content becomes excessively high. (The air void content increases with fiber content, and air voids tend to have a negative effect on many properties, such as the compressive strength.) In addition, the workability of the mix decreases with increasing fiber content. Moreover, the cost increases with increasing fiber content. Therefore, a rather low volume fraction of fibers is desirable. A fiber content as low as 0.2 vol% is effective, although fiber contents exceeding 1 vol% are common. The required fiber volume fraction increases with increasing fiber diameter and increases with increasing particle size of the aggregate.

The improvement in the structural properties due to the addition of discontinuous fibers to cement includes increases in the tensile ductility and flexural toughness and a decrease in the drying shrinkage. A low drying shrinkage is par-

ticularly valuable for large structures, as cracks can form due to the shrinkage and the cracks are wide for the same fractional shrinkage if the structure is large. In the case of the fiber being carbon fiber, improvements in the tensile strength and the flexural strength also occur. Carbon fibers (made from isotropic pitch) are advantageous in their superior ability to increase the tensile strength of cement, even though the tensile strength, modulus and ductility of the isotropic pitch based carbon fibers are low compared to most other fibers. Carbon fibers are also advantageous because of their relative chemical inertness.

In relation to most functional properties, carbon fibers are exceptional compared to the other fiber types. Carbon fibers are electrically conducting, in contrast to glass and polymer fibers, which are not conducting. Steel fibers are conductive, but their typical diameter ($\geq 60 \mu\text{m}$) is much larger than the diameter of a typical carbon fiber ($10 \mu\text{m}$). The combination of electrical conductivity and small diameter makes carbon fibers attractive for use in composite functional property tailoring.

1.3 Processing of Composite Materials

The technology and cost of composite materials depend largely on the processability; i.e., how the components are combined to form a composite material. The processability depends largely on the ability of the components to join, thereby forming a cohesive material. The processing often involves elevated temperatures and/or pressures. The required temperature and pressure, as well as the processing time, are typically dictated by the matrix material. The bonding of the filler with the matrix at an elevated temperature has a disadvantage in that bond weakening or even debonding may occur during the subsequent cooling, due to the difference in the thermal contraction (related to the coefficient of thermal expansion, or CTE) between filler and matrix. The bond weakening will result in the filler being less effective as a reinforcement, thus causing the mechanical properties of the composite to diminish. This problem tends to be particularly serious in metal-matrix composites, due to the relatively high processing temperatures involved.

Fiber composites are most commonly fabricated by the impregnation (or infiltration) of the matrix or matrix precursor in the liquid state into the fiber preform, which can take the form of a woven fabric. In the case of composites in the shape of tubes, the fibers may be impregnated in the form of a continuous bundle (called a tow) from a spool and subsequently the bundles can be wound on a mandrel. Instead of impregnation, the fibers and matrix material may be intermixed in the solid state by commingling reinforcing fibers and matrix fibers, by coating the reinforcing fibers with the matrix material, by sandwiching reinforcing fibers with foils of the matrix material, and in other ways. After impregnation or intermixing, consolidation is carried out, often under heat and pressure.

1.3.1 Polymer-Matrix Composites

Polymer-matrix composites (abbreviated PMC) can be classified according to whether the matrix is a thermoset or a thermoplastic polymer. Thermoset-

matrix composites are traditionally far more common, but thermoplastic-matrix composites are currently the focus of rapid development. The advantages of thermoplastic-matrix composites compared to thermoset-matrix composites include the following:

Lower manufacturing costs:

- No cure
- Unlimited shelf-life
- Reprocessing possible (for repair and recycling)
- Fewer health risks due to chemicals during processing
- Low moisture content
- Thermal shaping possible
- Weldability (fusion bonding possible).

Better performance:

- High toughness (damage tolerance)
- Good hot/wet properties
- High environmental tolerance.

The disadvantages of thermoplastic-matrix composites include the following:

- Limitations in relation to processing methods
- High processing temperatures
- High viscosities
- Prepreg (collection of continuous fibers aligned to form a sheet that has been impregnated with the polymer or polymer precursor) is stiff and dry when a solvent is not used (i.e., not drapeable or tacky)
- Fiber surface treatments less developed.

Fibrous polymer-matrix composites can be classified according to whether the fibers are short or continuous. Continuous fibers have much more effect than short fibers on the composite's mechanical properties, electrical resistivity, thermal conductivity, and on other properties too. However, they give rise to composites that are more anisotropic. Continuous fibers can be utilized in unidirectionally aligned tape or woven fabric form.

Polymer-matrix composites are much easier to fabricate than metal-matrix, carbon-matrix, and ceramic-matrix composites, whether the polymer is a thermoset or a thermoplastic. This is because of the relatively low processing temperatures required to fabricate polymer-matrix composites. For thermosets, such as epoxy, phenolic, and furfuryl resin, the processing temperature typically ranges from room temperature to about 200°C; for thermoplastic polymers, such as polyimide (PI), polyethersulfone (PES), polyetheretherketone (PEEK), polyetherimide

(PEI), and polyphenyl sulfide (PPS), the processing temperature typically ranges from 300 to 400°C.

Thermosets (especially epoxy) have long been used as polymer matrices for carbon fiber composites. During curing, usually performed in the presence of heat and pressure, a thermoset resin hardens gradually due to the completion of polymerization and the associated crosslinking of the polymer molecules. Thermoplastic polymers have recently become important because of their greater ductility and processing speed compared to thermosets, and the recent availability of thermoplastics that can withstand high temperatures. The higher processing speed of thermoplastics arises from the fact that amorphous thermoplastics soften immediately upon heating above the glass transition temperature (T_g), and so the softened material can be shaped easily. Subsequent cooling completes the processing. In contrast, the curing of a thermoset resin is a reaction that occurs gradually.

Short-fiber or particulate composites are usually fabricated by mixing the fibers or particles with a liquid resin to form a slurry, and then molding to form a composite. The liquid resin is the unpolymerized or partially polymerized matrix material in the case of a thermoset; it is the molten polymer or the polymer dissolved in a solvent in the case of a thermoplastic. The molding methods are those conventionally used for polymers by themselves. For thermoplastics, the methods include injection molding (heating above the melting temperature of the thermoplastic and forcing the slurry into a closed die opening through the use of a screw mechanism), extrusion (forcing the slurry through a die opening via a screw mechanism), calendering (pouring the slurry into a set of rollers with a small opening between adjacent rollers to form a thin sheet), and thermoforming (heating above the softening temperature of the thermoplastic and forming over a die using matching dies, a vacuum or air pressure, or without a die using movable rollers). For thermosets, compression molding or matched die molding (applying a high pressure and temperature to the slurry in a die to harden the thermoset) is commonly used. The casting of the slurry into a mold is not usually suitable because the difference in density between the resin and the fibers causes the fibers to float or sink unless the viscosity of the resin is carefully adjusted. To form a composite coating, the fiber-resin or particle-resin slurry can be sprayed instead of molded.

Instead of using a fiber-resin slurry, short fibers in the form of a mat or a continuous spun staple yarn can be impregnated with a resin and shaped using methods commonly used for continuous fiber composites. Yet another method involves using continuous staple yarns in the form of an intimate blend of short carbon fibers and short thermoplastic fibers. The yarns may be woven, if desired. They do not need to be impregnated with a resin to form a composite, as the thermoplastic fibers melt during consolidation under heat and pressure.

One method of forming unidirectional fiber composite parts with a constant cross-section (e.g., round, rectangular, pipe, plate, I-shaped) is pultrusion, in which fibers are drawn from spools, passed through a polymer resin bath for impregnation, and gathered together to produce a particular shape before entering a heated die.

Unidirectional composites suffer from poor mechanical properties in the transverse direction (i.e., perpendicular to the direction of pultrusion). Thus, structural composites are usually not unidirectional. Composites with continuous fibers in various orientations are commonly fabricated by hand lay-up of unidirectional fiber tapes or woven fabrics and impregnation with a resin. The molding, called bag molding, is done by placing the tapes or fabrics in a die and introducing high-pressure gases or a vacuum via a bag to force the individual plies together. Bag molding is widely used to fabricate large composite components for the skins of aircraft.

The fabrication of a high-performance polymer-matrix composite laminate containing continuous fibers commonly involves prepreg sheets. A prepreg is a sheet of continuous oriented fibers that have been impregnated with a polymer or a polymer precursor. An example of a polymer precursor is an epoxy resin, which upon subsequent curing (usually under heat and pressure) forms epoxy polymer, a thermoset. Figure 1.2 shows a scanning electron microscope image of the surface of a carbon fiber epoxy prepreg. The fibers cannot be discerned because they are covered with the resin. An example of a polymer in a prepreg is nylon, which is a thermoplastic polymer. The softening of the thermoplastic polymer upon heating above the glass transition temperature allows the prepreg to be flexible, thus conforming to the desired shape. In the case of an epoxy resin prepreg, the prepreg is flexible and tacky as long as the resin has not been cured. In order to increase the usable period of an epoxy resin prepreg, the prepreg is typically stored in a freezer, as the low temperature in the freezer helps slow down the curing process. Even with storage in a freezer, the usable period of an epoxy resin prepreg is only a few months.

Instead of using unidirectional fibers, one can use a woven fiber fabric. The fabric may be impregnated with the resin or the polymer prior to being stacked

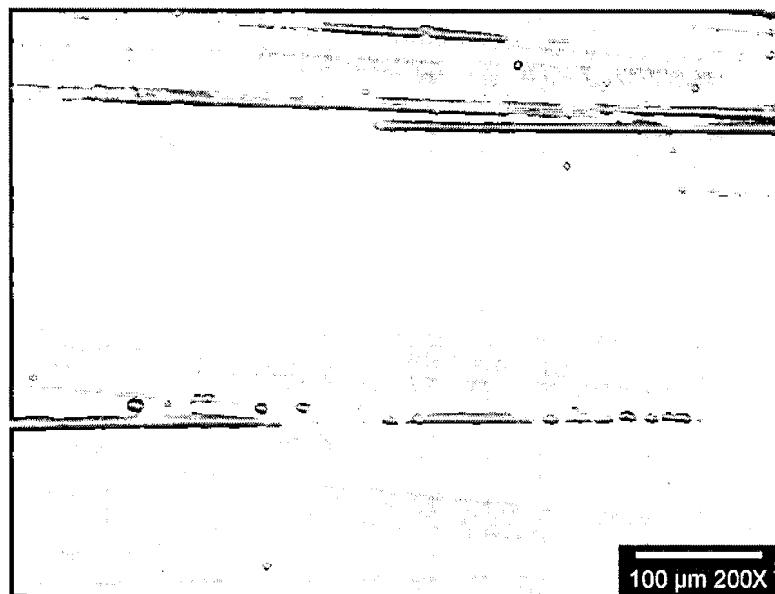


Figure 1.2. A scanning electron microscope image of the surface of a carbon fiber epoxy prepreg. The fibers cannot be discerned due to their being covered with the resin

and consolidated to form a composite. Related to weaving is braiding, which is commonly used to form a composite tubing.

The fabric may be stacked in the absence of a resin and then the resin is infiltrated into the stack – a process known as resin transfer molding (RTM). The advantage of using a fabric is that fabrics are easy to handle. RTM is attractive in that it allows the fabrication of composites of intricate shapes. In RTM, a fiber preform (usually prepared by weaving or braiding and held under compression in a mold) is impregnated with a resin. The resin is admitted at one end of the mold and is forced by pressure through the mold and preform. The resin is subsequently cured. This method is limited to resins of low viscosity, such as epoxy. A problem with this process is the formation of surface voids by the volatilization of dissolved gases in the resin, the partial evaporation of mold releasing agent into the preform, or the mechanical entrapment of gas bubbles.

The weaving of fibers to form a fabric results in the local bending of the fibers in the resulting fabric. The bending occurs where a fiber in one direction crosses that in another direction, as illustrated in Fig. 1.3. Due to this bending, the mechanical properties of a composite made from fabric tend to be inferior to those of a composite made from unidirectional preregs.

Most of the composite fabrication methods mentioned above involve the impregnation of the fibers with a resin. In the case of a thermoset, the resin is a liquid that has not been polymerized or is partially polymerized. In the case of a thermoplastic, the resin is either the polymer melt or the polymer dissolved in the solvent. After resin application, solid thermoplastic results from solidification in the case of melt impregnation, and from evaporation in the case of solution impregnation. Both amorphous and semicrystalline thermoplastics can be melt processed, but only the amorphous resins can normally be dissolved. Because of the high melt viscosities of semicrystalline thermoplastics (due to their long and rigid macromolecular chains), direct melt impregnation of semicrystalline thermoplastics is difficult. Melt impregnation followed by solidification produces a thermoplastic prepreg that is stiff and lacks tack; solution impregnation usually produces preregs that are drapeable and tacky, although this character changes as solvent evaporation occurs from the solution. The drapeable and tacky character of thermoplastic preregs made by solution impregnation is comparable to that of thermoset preregs. Hence, the main problem with resin impregnation occurs for semicrystalline thermoplastics. Instead of thermoplastic impregnation of fibers by using a melt or a solution of the thermoplastic, solid thermoplastic in the form of powder, fibers, or slurries can be impregnated.

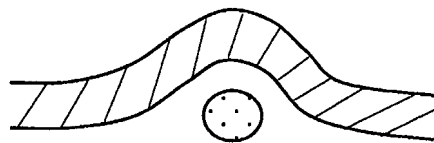


Figure 1.3. The bending of a fiber in a woven fabric. The circle represents a fiber that is perpendicular to the paper. The other paper, which is in the plane of the paper, bends around it

An alternative to impregnation is the commingling of continuous reinforcing fibers with continuous thermoplastic fibers. This commingling can be performed on a fabric level, where yarns of different materials are woven together (coveaving); it can be performed on a yarn level, where yarns of different materials are twisted together; or it can be performed on a fiber level, where fibers of different materials are intimately mixed within a unidirectional fiber bundle. During processing (such as compression molding or filament winding), the thermoplastic fibers melt, wet the fibers, and fuse to form the matrix. However, there is a preferred orientation in the thermoplastic fibers due to the spinning process used in their production, and this may be a problem. Furthermore, the thermoplastic fibers have a tendency to form drops during heating. In addition, the availability of high-temperature thermoplastic fibers is limited. PEEK is most commonly used for commingling with carbon fibers. Fiber-matrix adhesion in a commingled system depends on the molding temperature, residence time at the melt temperature, and the cooling rate. This is probably due to several complex mechanisms such as matrix adsorption on the fiber surface, matrix degradation leading to chemical bonding, and interfacial crystallization. On the other hand, prepregs made from commingled fibers are flexible and drapeable, and the use of three-dimensional braiding allows net structural shape formation and enhances damage tolerance due to the lack of delamination. To prevent the ends of the braided preform from unbraiding, the thermoplastic fibers are melted with a soldering gun before cutting, or alternately the ends of the braided preform are wrapped with a polyimide tape and cut through the tape. The fiber commingling makes a uniform polymer distribution possible even when the three-dimensional preform is very large, although the heating time during consolidation needs to be longer for dense three-dimensional commingled fiber network braids than for unidirectional prepregs.

The shaping of thermoplastic-matrix composite laminates can be performed by thermoforming in the form of matched-die forming or die-less forming. However, in addition to shaping, deformations in the form of transverse fiber flow (shear flow perpendicular to the fiber axis) and interply slip commonly occur, while intraply slip is less prevalent. Die-less forming uses an adjustable array of universal, computer-controlled rollers to form an initially flat composite material into a long, singly curved part with one arbitrary cross-sectional shape at one end and another arbitrary shape at the other end. Heating and bending of the material are strictly local processes, occurring only within a small active forming zone at any one instant. The initially flat workpiece is translated back and forth along its length in a number of passes. On successive passes, successive portions of the transverse extent of the workpiece pass through the active forming region. Induction heating is used to provide the local heating in die-less forming, because it enables rapid, noncontact, localized and uniform through-thickness heating.

The schedule for varying the temperature and pressure during the curing and consolidation of prepregs to form a thermoset-matrix composite must be carefully controlled. Curing refers to the polymerization and crosslinking relations that occur upon heating and lead to the polymer, whereas consolidation refers to the application of pressure to obtain proper fiber-matrix bonding, low void content,

and the final shape of the part. Curing and consolidation are usually performed together as one process.

One method of forming continuous fiber composites in the shape of cylinders or related objects is filament winding, which involves wrapping continuous fibers from a spool around a (commonly cylindrical) mandrel. The fibers are wound in various predetermined directions (e.g., 90°) relative to the axis of the mandrel. The winding pattern is a part of the composite design. Since the composite is very strong in the fiber direction, filament winding results in a cylindrical article that resists radial expansion, as needed for pressure vessels. The fibers can be impregnated with a resin before or after winding. Filament winding is used to make pressure tanks. The temperature of the mandrel, the impregnation temperature of the resin, the impregnation time, the tension of the fibers, and the pressure of the fiber winding are processing parameters that need to be controlled.

The processing of polymer-matrix composites typically requires heating. In the case of a thermosetting resin, the heating is to cause the completion of polymerization (cross-linking) of the resin. In the case of a thermoplastic matrix, the heating is done to soften or melt the thermoplastic matrix. (The melting temperature is higher than the softening temperature but it allows more extensive flow.) As the polymerization process is a reaction, it takes time. In contrast, the softening or melting is a phase transition that occurs once the appropriate temperature is reached. As a result, the processing time tends to be considerably longer for a thermoset-matrix composite than a thermoplastic-matrix composite.

1.3.2 Metal-Matrix Composites

The processing of a metal-matrix composite (abbreviated MMC) tends to be much more expensive than that of a polymer-matrix composite due to the high processing temperature required. The fabrication of metal-matrix composites often involves the use of an intermediate, called a preform, in the form of sheets, wires, cylinders, or near-net shapes. The size and shape of the preform are the same as those of the composite article to be made. The preform contains the reinforcement, which is usually held together by a binder that can be a polymer (e.g., acrylic, styrene), a ceramic (e.g., silica, aluminum metaphosphate), or the matrix metal itself. For example, continuous fibers are wound around a drum and bound with a resin, and subsequently the wound fiber cylinder is cut off the drum and stretched out to form a sheet. During subsequent composite fabrication, the organic binder evaporates. As another example, short fibers are combined with a ceramic or polymeric binder and a liquid carrier to form a slurry; this is then filtered under pressure or wet pressed to form a wet "cake," which is subsequently dried to form a preform. When the matrix metal is used as the binder, a continuous fiber bundle is immersed in the molten matrix metal so as to be infiltrated with it, thus forming a wire preform; alternately, fibers placed on a matrix metal foil are covered and fixed in place with a sprayed matrix metal, thus forming a sprayed preform.

A binder is not always needed, although it helps the fibers to stay uniformly distributed during subsequent composite fabrication. Excessive ceramic binder

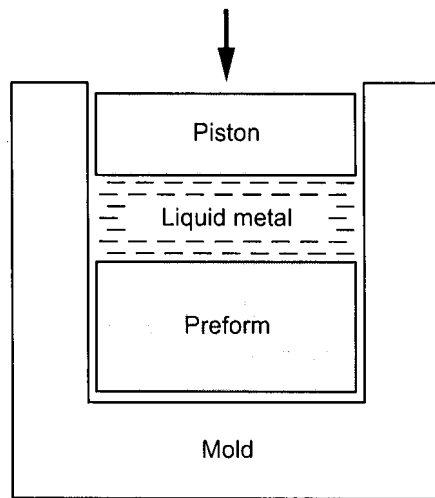


Figure 1.4. Process of liquid metal infiltration for the fabrication of a metal-matrix composite

amounts should be avoided, as they can make the resulting metal-matrix composite more brittle. For ceramic binders, a typical amount ranges from 1 to 5 wt.% of the preform. When woven fabrics are used as reinforcement, a binder is less important, as the weaving itself serves to hold the fibers together in a uniform fashion.

The most popular method of fabricating metal-matrix composites is the infiltration of a preform by a liquid metal, typically under pressure, as illustrated in Fig. 1.4. The temperature needs to be above the solidus temperature (i.e., the temperature above which liquid metal exists, such that the liquid metal coexists with the solid metal unless the temperature is above the liquidus). In order to use a liquid metal infiltration temperature that is not too high, a metal (e.g., aluminum) with a relatively low melting temperature is usually chosen for the matrix.

The low viscosities of liquid metals compared to resins or glasses make infiltration very appropriate for metal-matrix composites. Nevertheless, pressure is usually required because it is difficult for the liquid metal to wet the reinforcement. The pressure can be provided by a gas (e.g., argon) or a piston, as illustrated in Fig. 1.4. When a piston is used, the process can be quite fast and is known as squeeze casting. The lower the pressure, the higher the porosity in the resulting composite, as shown in Fig. 1.5. In addition, the higher the filler volume fraction, the higher the porosity tends to be, as also shown in Fig. 1.5.

One challenge when using liquid metal infiltration to fabricate particulate metal-matrix composites relates to the difficulty involved in making composites with a low volume fraction of the filler due to how difficult it is to make preforms with a low volume fraction of filler. This difficulty stems from the fact that filler particles naturally contact one another in the form. One method of achieving a low filler volume fraction involves using a combustible secondary particulate (e.g., carbon particles) in addition to the noncombustible filler (e.g., silicon carbide particles) in the preform. After preform fabrication, the combustible particulate is removed by oxidation (i.e., burning), thus resulting in a preform with a low filler volume fraction (as low as 18%). Figure 1.6 shows SEM photographs of a preform with

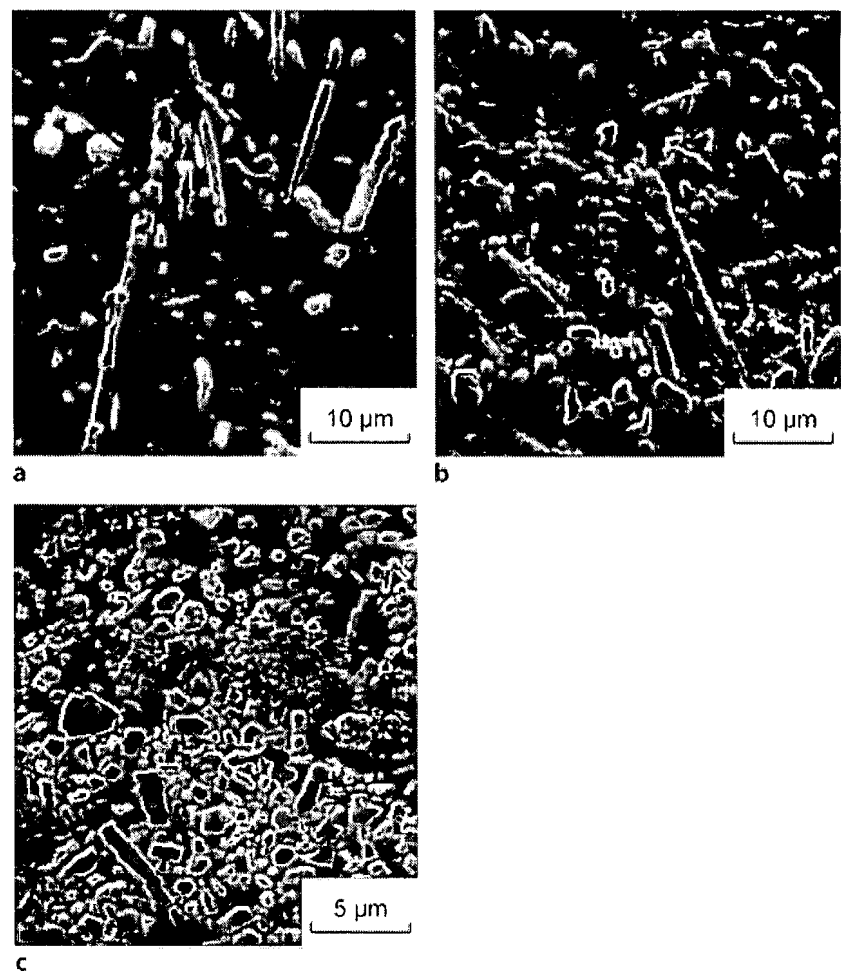


Figure 1.5. Scanning electron microscope photographs of polished sections of aluminum-matrix silicon carbide whisker composites fabricated by liquid metal infiltration. The whiskers have diameter 1.4 μm and length 18.6 μm . **a** 10 vol% SiC whiskers, with infiltration pressure of 1.4 MPa and porosity 5%. **b** 10 vol% SiC whiskers, with infiltration pressure of 13.8 MPa and porosity <0.5%. **c** 31 vol% SiC whiskers, with infiltration pressure of 13.8 MPa and porosity 3%. (From [2])

silicon carbide particles (3–5 μm) used as the reinforcement and carbon particles (activated carbon, equiaxed, 20 μm) as the combustible particulate before and after burning the carbon [1].

The infiltration method can be used to produce near-net shape composites, so that subsequent shaping is not necessary. As infiltration using a gas pressure involves a smaller rate of pressure increase compared to using a piston, infiltration is more suitable than squeeze casting for near-net shape processing. If shaping is necessary, it can be achieved by plastic forming (e.g., extrusion, swaging, forging, and rolling) when short fibers or particles are used as the reinforcement. Plastic forming tends to reduce the porosity and give a preferential orientation to the short fibers. These effects result in improved mechanical properties. For continuous fiber metal-matrix composites, shaping cannot be achieved by plastic forming, and cutting is necessary.

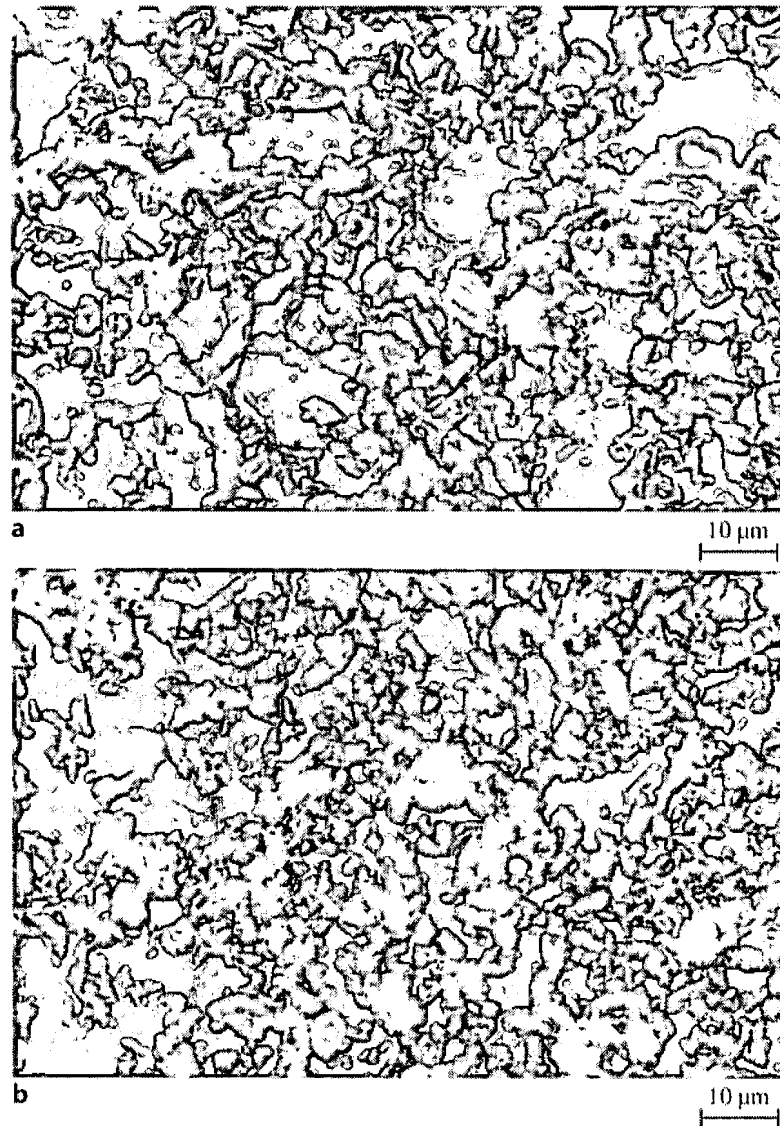


Figure 1.6. SEM photographs of a preform with SiC particles (reinforcement) and carbon particles (combustible particulate), fabricated using an acid phosphate binder. **a** Before burning off the carbon particles (dark large particles). **b** After burning off the carbon particles. (From [1])

A second method for fabricating metal-matrix composites is diffusion bonding. In this method, a stack of alternating layers of fibers and metal foils is hot pressed (at, say, 24 MPa for 20 min) to cause bonding in the solid state. This method is not very suitable for fiber cloths or continuous fiber bundles because it is difficult for the metal to flow into the space between the fibers during diffusion bonding. In contrast, the infiltration method involves melting the metal, so metal flow is relatively easy, making infiltration a more suitable method for fiber cloths or continuous fiber bundles. A variation of diffusion bonding involves the hot pressing of metal-coated fibers without the use of metal foils. In this case, the metal coating provides the metal for the metal-matrix composite. In general, the

diffusion bonding method is complicated by the fact that the surface of the metal foil or metal coating tends to be oxidized and the oxide makes the bonding more difficult. Hence, a vacuum is usually required for diffusion bonding.

A third method of fabricating metal-matrix composites involves hot pressing above the solidus of the matrix metal. This method requires lower pressures than diffusion bonding, but the higher temperature of the pressing tends to cause reinforcement degradation, resulting from the interfacial reaction between the reinforcement and the matrix metal. One way to alleviate this problem is to insert a metal sheet of a lower solidus than the matrix metal alternately between the wire preform layers and then hot press at a temperature between the two solidus temperatures.

A combination of the second and third methods involves first heating fibers laid up with matrix metal sheets between them in vacuum in a sealed metal container above the liquidus of the matrix metal, then immediately hot pressing the container at a temperature below the solidus of the matrix metal.

A fourth method of fabricating metal-matrix composites involves the plasma spraying of the metal onto continuous fibers. As this process usually results in a composite of high porosity, subsequent consolidation (e.g., by hot isostatic pressing) is usually necessary. Compared to the other methods, plasma spraying has the advantage of being able to produce continuous composite parts, though the subsequent consolidation step may limit their size.

Slurry casting, a fifth method, is complicated by the tendency for the carbon fibers (low in density) to float on the metal melt. To overcome this problem, which causes nonuniformity in the fiber distribution, compocasting is necessary. Compocasting (rheocasting) involves vigorously agitating a semi-solid alloy so that the primary phase is nondendritic, thereby giving a fiber-alloy slurry with thixotropic properties.

A sixth method of making a metal-matrix composite is power metallurgy (PM). This process involves the sintering of the metal matrix particles when these particles have been mixed with the filler (Fig. 1.7). The sintering involves solid-state diffusion, which causes the metal particles to join to one another. The joining starts with the formation of a neck between two adjacent particles, as illustrated in Fig. 1.8. As sintering progresses, the necks become wider, so that the porosity in the overall material becomes smaller. Because the diffusion is in the solid state, the temperature is below the melting temperature of the metal. However,

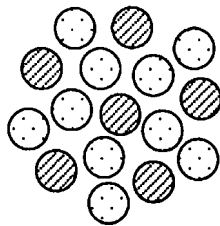


Figure 1.7. A mixture of the filler particles and the metal particles, as used in the admixture method of powder metallurgy for the fabrication of a metal-matrix composite

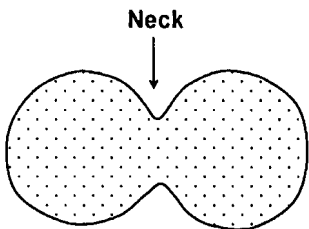


Figure 1.8. The formation of a neck between two metal particles during the fabrication of a metal-matrix composite by powder metallurgy

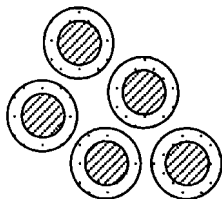


Figure 1.9. Filler particles coated with metal for making a metal-matrix composite by the coated filler method of powder metallurgy

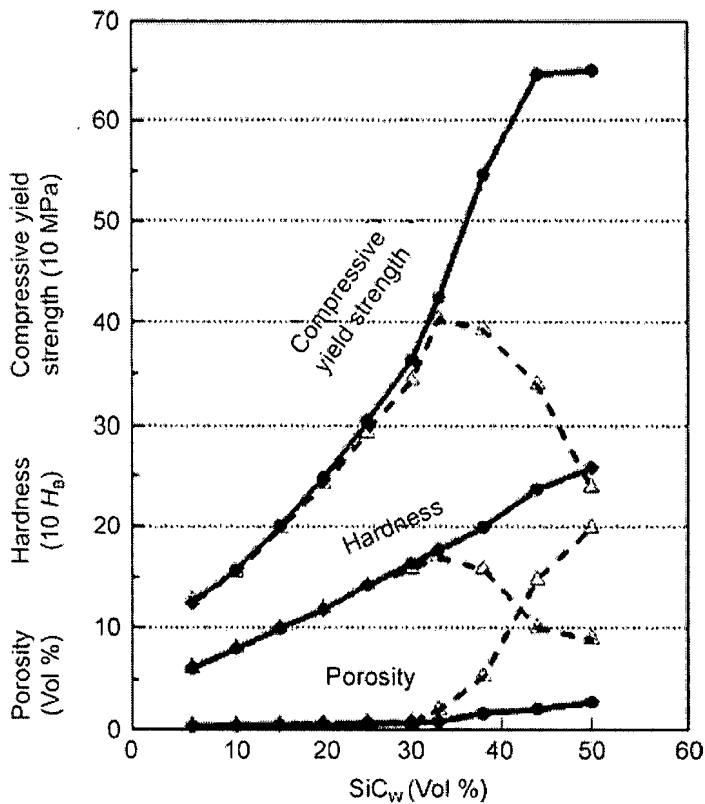


Figure 1.10. Variation in the porosity, hardness and compressive yield strength of silicon carbide whisker copper-matrix composites fabricated by powder metallurgy. *Circles*, coated filler method. *Triangles*, admixture method. (From [3])

the temperature must be adequate for the diffusion to be significant. This process requires not only high temperature but also high pressure, since pressure helps the densification process (i.e., the reduction of the porosity of the article subjected to sintering). The pressure may be applied before or during the sintering. Due to the need for high pressure, an article made by this method is limited in size. Since the melting temperature of the filler is usually much higher than that of the metal matrix, the sintering pertains to the metal particles rather than to the filler. As a result, the volume fraction of filler in the resulting composite is usually low (e.g., 20%); otherwise there is not enough metal matrix to bind the filler particles (or fibers) together to form a composite. To alleviate this problem, the filler particles (or fibers) may be coated with the metal matrix (e.g., by plating) prior to sintering (Fig. 1.9). By using the coated filler, with the use of the metal particles being optional, the filler volume fraction can be greatly increased compared to when the uncoated filler is used. A high filler volume fraction (e.g., 60%) is necessary to achieve a low CTE, as needed for microelectronic heat sinks. Just for mechanical properties, a high filler volume fraction is not necessary.

Figure 1.10 shows that the advantage of the coated filler method over the conventional admixture method is particularly significant when the filler volume fraction is high (e.g., more than 30 vol% when silicon carbide whisker is used as the filler).

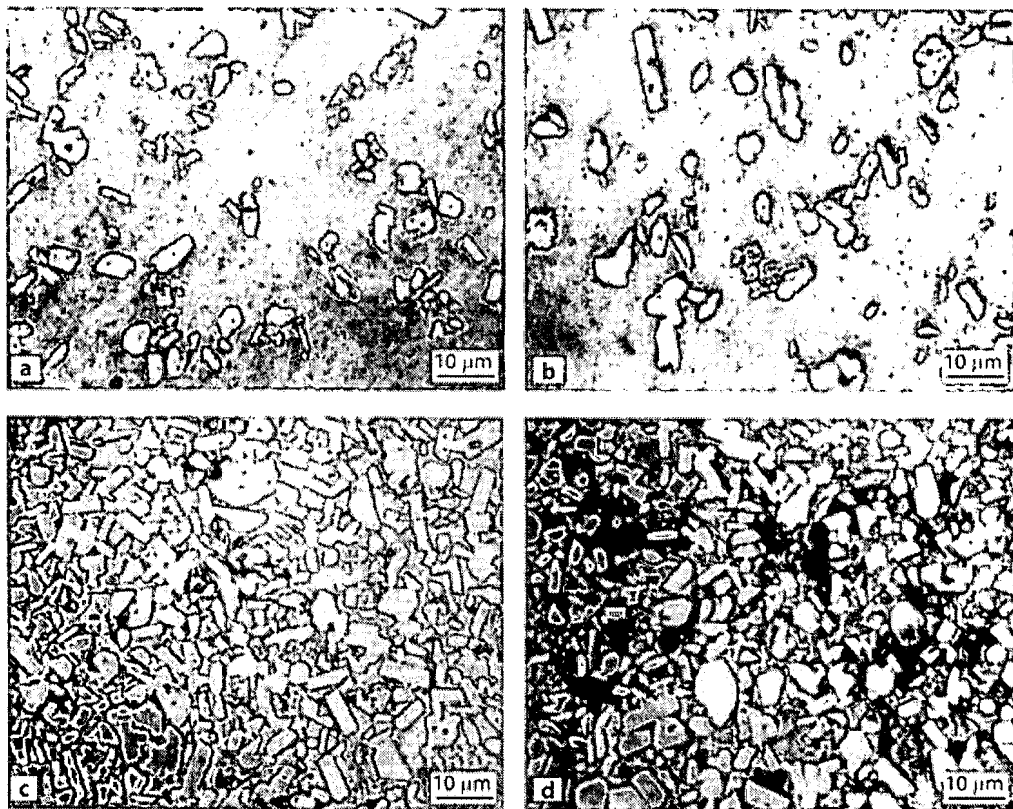


Figure 1.11. Optical microscope photographs of copper-matrix titanium diboride platelet composites fabricated by powder metallurgy. **a** 15 vol% TiB_2 , coated filler method; **b** 15 vol% TiB_2 , admixture method; **c** 60 vol% TiB_2 , coated filler method; **d** 60 vol% TiB_2 , admixture method. (From [4])

In the regime of high filler volume fraction, the coated filler method gives lower porosity, higher hardness and higher compressive yield strength. Due to its high modulus and high aspect ratio, silicon carbide whisker is a highly effective reinforcement. Silicon carbide particles are commonly used as an abrasive. Figure 1.11 shows copper-matrix composites made by the two methods and containing 15 vol% titanium diboride (TiB_2) platelets ($3\text{--}5\text{ }\mu\text{m}$) have similarly low porosities, but that the composite made using the admixture method has a much higher porosity than that made using the coated filler method when the composite contains 60 vol% TiB_2 platelets. Titanium diboride is a ceramic that is unusual in its high thermal conductivity (about 100 W/m K) and low electrical resistivity (about $20 \times 10^{-6}\text{ }\Omega\text{ cm}$).

1.3.3 Carbon-Matrix Composites

Carbon-matrix composites are made by the carbonization of the matrix of a polymer-matrix composite. The process of carbonization (also known as pyrolysis) is akin to charring, which involves a chemical reaction that removes essentially all atoms that are not carbon from the polymer (the carbon precursor). The carbonization process is typically conducted in an inert atmosphere at around $650\text{--}1,200^\circ\text{C}$. The atmosphere is usually nitrogen at temperatures up to $1,000^\circ\text{C}$ and is argon at temperatures above $1,000^\circ\text{C}$. Argon at high temperatures is used because carbon tends to react with nitrogen to form poisonous cyanide compounds above $1,000^\circ\text{C}$. One common carbon precursor is pitch (a polyaromatic hydrocarbon material derived from petroleum or coal), which is a mixture of thermoplastic polymers. Pitch is attractive because of its relatively high char yield (i.e., the mass of the resulting carbon divided by the mass of the carbon precursor). A high char yield means a smaller amount of porosity in the resulting carbon-matrix composite. There are many grades of pitch. Those with higher molecular weights tend to give higher char yields. Due to the porosity, which is detrimental to the mechanical properties of the composite, the carbon-matrix composite is commonly subjected to impregnation of the carbon precursor and then another round of carbonization. In order to achieve a sufficiently low porosity, multiple cycles of impregnation-carbonization are conducted, thus resulting in a high processing cost. In order to decrease the required number of cycles, the last cycle is commonly followed by a process known as chemical vapor infiltration (CVI), in which the remaining pores are filled with carbon that is deposited from the thermal decomposition of a carbonaceous gas that infiltrates the composite.

A thermoplastic polymer such as pitch melts upon heating. The melting of the matrix is undesirable for the conversion of a polymer-matrix composite to a carbon-matrix composite. Furthermore, during carbonization, pitch tends to bloat due to the evolution of gases generated by pyrolysis. The bloating can cause the expulsion of pitch from the carbon fiber preform during carbonization. Thus, the carbonization process is usually preceded by a process known as stabilization, which involves heating at $200\text{--}300^\circ\text{C}$ in the presence of oxygen. In stabilization, oxygen diffuses into the carbon precursor and reacts with it, thus converting it

chemically to a form that does not melt. Hence, the stabilization process is also known as infusibilization.

The fabrication of carbon-carbon composites is carried out using four main methods, namely:

1. liquid phase impregnation (LPI);
2. hot isostatic pressure impregnation carbonization (HIPIC);
3. hot pressing, and;
4. chemical vapor infiltration (CVI).

All of these methods (except, in some cases, CVI) involve the initial preparation of a prepreg by either wet winding continuous carbon fibers with pitch or resin (e.g., phenolic), or wetting woven carbon fiber fabrics with pitch or resin. Unidirectional carbon fiber tapes are not as commonly used as woven fabrics, because fabric lay-ups tend to result in more interlocking between the plies. For highly directional carbon-carbon composites, fabrics that have a greater number of fibers in the warp direction than the fill direction may be used. After prepreg preparation and, in the case of fabrics, fabric lay-up, the pitch or resin needs to be pyrolyzed or carbonized by heating at 350–850°C. Due to the shrinkage of the pitch or resin during carbonization (which is accompanied by the evolution of volatiles), additional pitch or resin is impregnated in the case of LPI and HIPIC, and carbonization is carried out under pressure in the case of hot pressing. In LPI, carbonization and impregnation are carried out as distinct steps; in HIPIC, carbonization and impregnation are performed together as a single step.

The carbon yield (or char yield) from carbonization is around 50 wt.% for ordinary pitch and 80–88 wt.% for mesophase pitch at atmospheric pressure. Mesophase pitch tends to be more viscous than ordinary pitch, making impregnation more difficult. In the case of resins, the carbon yield varies widely from one resin to another. Significant increases in the carbon yield of pitch can be obtained through the use of high pressure during carbonization. Mesophase spheres that exhibit a highly oriented structure similar to liquid crystals and are initially around 0.1 μm in diameter are observed in isotropic liquid pitch above 400°C. Prolonged heating causes the spheres to coalesce, solidify, and form larger regions with lamellar order; this favors graphitization upon subsequent heating to $\sim 2,500^\circ\text{C}$. The high pressure during carbonization lowers the temperature at which the mesophase forms. At very high pressures (~ 200 MPa), coalescence of mesophase does not occur. Therefore, an optimum pressure is around 100 MPa. Pressure may or may not be applied during carbonization in LPI, but it is always applied during carbonization in HIPIC.

In LPI, after carbonization, vacuum impregnation is performed with additional pitch or resin in order to densify the composite. Pressure (e.g., 2 MPa) may be applied to help the impregnation. The first carbonization decreases the density from the value of the green composite, so that subsequent impregnation and re-carbonization are necessary. The carbonization-impregnation cycles are repeated several times (typically 3–6) in order to achieve sufficient densification. Both the density and interlaminar shear strength (ILSS) increase with the number of cycles.

The density levels off after a few cycles of impregnation and recarbonization. This is because the repeated densification cycles cause the mouths of the pores to narrow down, making it difficult for the impregnant to enter the pores. As a consequence, impregnant pickup levels off. This problem can be alleviated by intermediate graphitization, where the composites are subjected to heat treatment at 2,200–3,000°C between the carbonization and impregnation steps after the densification cycle when the density levels off. On graphitization, the pore entrances open up due to the rearrangement of the crystallites in the matrix. These opened pores then become accessible during further impregnation, thus leading to a further increase in density. The use of mesophase pitch instead of isotropic pitch for impregnation can cut down on the required number of impregnation cycles.

In HIPIC, an isostatic inert gas pressure of around 100 MPa is applied to impregnate pitch (rather than resins, which suffer from a low carbon yield) into the pores in the sample while the sample is being carbonized at 650–1,000°C. The pressure increases the carbon yield and maintains the more volatile fractions of the pitch in a condensed phase. After this combined step of carbonization and impregnation, graphitization is performed by heating without applied pressure above 2,200°C. HIPIC allows the density to reach a higher value than LPI (with or without intermediate graphitization), and fewer cycles are needed for HIPIC to achieve the high density. However, HIPIC is an expensive technique.

One HIPIC process involves vacuum impregnating a dry fiber preform or porous carbon–carbon laminate with molten pitch by placing it inside a metal container (or can) containing an excess of pitch. The can is then evacuated and sealed (preferably using an electron beam weld) and placed within the work zone of a hot isostatic press (HIP) unit. The temperature is then raised at a programmed rate above the melting point of the pitch, but not high enough to result in weight loss due to the onset of carbonization. The pressure is then increased and maintained at around 100 MPa. The pitch initially melts and expands within the can and is forced by isostatic pressure into the pores in the sample. The sealed container acts like a rubber bag, facilitating the transfer of pressure to the workpiece. After that, the temperature is gradually increased towards that required for pitch carbonization (650–1,000°C). The pressure not only increases the carbon yield but also prevents liquid from being forced out of the pores by pyrolysis products. The optimum carbonization pressure for HIPIC is 100–150 MPa. Lower pressures are insufficient to prevent bloating of the composites due to the evolution of carbonization gases.

HIPIC increases the carbon yield of pitch, especially when the molecular size of the pitch is small. The increase in pressure up to 10 MPa causes the carbon yield to increase significantly for a pitch with a low molecular weight, but only slightly for a pitch with a high molecular weight. This is due to the already high carbon yield of pitch with a high molecular weight when the pressure is low. This improvement can be attributed to the trapping and decomposition of the evolved hydrocarbon gases under high pressure; the decomposition produces carbon and hydrogen. The increase in pressure causes the bulk density to increase, the porosity to decrease, and the flexural strength to increase.

In hot pressing (also called high-temperature consolidation), carbonization is performed at an elevated temperature (1,000°C typically) under uniaxial pressure

(2–3 MPa typically) in an inert or reducing atmosphere or in a vacuum. During hot pressing, graphitization may occur even for thermosetting resins, which are harder to graphitize than pitch. This is known as stress graphitization. Subsequently, further graphitization may be performed by heating without applied pressure at 2,200–3,000°C. No impregnation is performed after the carbonization. Composites made by hot pressing have flattened pores in the carbon matrix, and the part thickness is reduced by about 50%.

In CVI (also called CVD, chemical vapor deposition), gas phase impregnation of a hydrocarbon gas (e.g., methane, propylene) into a carbon fiber preform takes place at 700–2,000°C, so that pyrolytic carbon produced by the cracking of the gas is deposited in the open pores and surface of the preform. The carbon fiber preform can take the form of carbon fabric prepregs that have been carbonized and graphitized, or the form of dry wound carbon fibers. There are three CVI methods, namely the isothermal method, the temperature gradient method, and the pressure gradient method.

In the isothermal method, the gas and sample are kept at a uniform temperature. As carbon growth in the pores will cease when they become blocked, there is a tendency for preferential deposition on the exterior surfaces of the sample. This results in a need for multiple infiltration cycles such that the sample is either skinned by light machining or exposed to high temperatures to reopen the surface pores for more infiltration in subsequent cycles.

In the temperature gradient method, an induction furnace is used. The sample is supported by an inductively heated mandrel (a susceptor) so that the inside surface of the sample is at a higher temperature than the outside surface. The hydrocarbon gas flows along the outside surface of the sample. Due to the temperature gradient, the deposition occurs first at the inside surface of the sample and progresses toward the outside surface, thereby avoiding the crusting problem.

In the pressure gradient method, the hydrocarbon gas impinges on the inside surface of the sample, so the gas pressure is higher at the inside surface than the outside surface. The pressure gradient method is not as widely used as the isothermal method or the temperature gradient method.

Both the temperature gradient method and the pressure gradient method are limited to single samples, whereas the isothermal method can handle several samples at once. However, the isothermal method is limited to thin samples due to the crusting problem.

A drawback of CVI is the low rate of deposition resulting from the use of a low gas pressure (1–150 Torr), which favors a long mean free path for the reactant and decomposed gases; a long mean free path enhances deposition into the center of the sample. A diluent gas (e.g., He, Ar) is usually used to help the infiltration. Hydrogen is often used as a carbon surface detergent.

An attraction of CVI is that CVI carbon is harder than char carbon from pitch or resin, making CVI carbon particularly desirable for carbon–carbon composites used for brakes and friction products.

The fiber–matrix bond strength in carbon–carbon composites must be optimal. If the bond strength is too high, the resulting composite may be extremely brit-

tle and thus exhibit catastrophic failure and poor strength. If it is too low, the composites fail in pure shear, with poor transfer of the load to the fiber.

The main disadvantages of carbon-carbon composites are their high fabrication cost, poor oxidation resistance, poor interlaminar properties (especially for two-dimensionally woven fibers), the difficulty involved in making joints, and the insufficient engineering database.

1.3.4 Ceramic-Matrix Composites

A ceramic-matrix composite (abbreviated to CMC) is commonly made from a polymer-matrix composite by converting the polymer matrix to a ceramic. The process often involves thermal composition and, in this case, it is known as pyrolysis. For example, polycarbosilane (a polymer that has both carbon and silicon atoms) is often used as the precursor of silicon carbide.

The fibers can be short or continuous. In both cases, the composites can be formed by viscous glass consolidation, i.e., either hot pressing a mixture of fibers and glass powder, or by winding glass-impregnated continuous fiber under tension above the annealing temperature of the glass.

Short (≤ 3 mm) fiber borosilicate glass composites are prepared by hot pressing, in vacuum or argon, using an isopropyl alcohol slurry of fibers and Pyrex powder ($< 50 \mu\text{m}$ particle size) at $700\text{--}1,000^\circ\text{C}$ (depending on the fiber content, which ranges from 10 to 40 vol%) and 6.9 MPa. Fibers longer than 3 mm are more difficult to disperse in the slurry. The resulting composite has fibers that are randomized two-dimensionally, as some alignment occurs during pressing.

Continuous fiber composites are made by allowing fibers from a spool to pass through a glass powder slurry (containing water and a water-soluble acrylic binder), winding the slurry-impregnated fibers onto the sides of a hexagonal prism (mandrel or take-up drum), cutting up the flat unidirectional tapes from the mandrel, stacking the pieces (plies) in a proper orientation, burning out the stack to remove the binder, and hot pressing the stack at a temperature above the working temperature of the glass. This process is known as slurry infiltration or viscous glass consolidation. During hot pressing, the glass must flow into the space between adjacent fibers. Since glass does not wet carbon, sufficient pressure is necessary.

Viscous glass consolidation can also be carried out without hot pressing. In this case, a glass powder-impregnated continuous fiber tow is wound under tension (e.g., about 15 ksi or 100 MPa) onto a collection mandrel at a temperature above the annealing temperature of the glass.

Other than viscous glass consolidation, another method for forming short or continuous fiber glass-matrix composite is the sol-gel method, i.e., infiltration by a sol and subsequent sintering, using metal alkoxides and/or metal salts as precursors and using an acid or base catalyst to promote hydrolysis and polymerization. Composite fabrication consists of two steps:

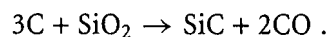
1. the preparation of fiber-gel prepregs, and
2. thermal treatment and densification by hot pressing.

In the first step, the sol-impregnated prepreps are allowed to gel, dried at room temperature for a day, dried at 50°C for a day, and then heat-treated at 300–400°C for 3 h. In the second step, hot pressing is performed in a nitrogen atmosphere with a pressure of 10 MPa. In the case of a borosilicate glass, the hot pressing temperature is between 900 and 1,200°C. The sol-gel method is advantageous in that homogenization of the components during impregnation can readily be achieved and sintering temperatures are substantially lowered because of the smaller particle size compared to the slurry infiltration method. The easier impregnation or infiltration decreases the tendency for the particles to damage the fibers. However, the sol-gel method has the disadvantage of excessive shrinkage during subsequent heat treatment.

The slurry infiltration and sol-gel methods can be combined using particle-filled sols. Particle filling helps to reduce the shrinkage.

Yet another method is melt infiltration, which requires that the glass is heated at a temperature much above the softening temperature in order for the glass to infiltrate the fiber preform. This high temperature may lead to a chemical reaction between the fibers and the matrix.

The reaction between SiO_2 and carbon fibers is of the form:



To reduce the extent of this reaction, SiC-coated carbon fibers are used for SiO_2 -matrix composites.

Fibers such as carbon fibers have the following effects on the glass:

1. they increase the toughness (and, in some cases, the strength as well);
2. they decrease the coefficient of thermal expansion, and;
3. they increase the thermal conductivity.

The increase in toughness occurs in glass with short and randomly oriented fibers, as well as glass with continuous fibers. However, the strength is usually decreased by the fibers in the former and is usually increased by the fibers in the latter. Even for continuous fibers, the strengthening is limited by widespread matrix cracking (both transverse and longitudinal).

The high-temperature strength of carbon fiber reinforced glasses in air is limited by the oxidation of the carbon fibers. This oxidation cannot be prevented by the glass matrix. In an inert atmosphere, the high-temperature strength is limited by the softening of the glass matrix.

1.3.5 Cement-Matrix Composites

Cement is basically calcium silicate, where the SiO_4 tetrahedron (with the silicon atom being sp^3 hybridized and located at the body center of the tetrahedron, while the oxygen atoms are located at the four corners of the tetrahedron) is the fundamental building block. The realization of a cement-matrix composite does not require heat or pressure. This is why concrete processing can be performed in the

field (as in road construction) rather than in a factory. The process involves mixing cement particles with water and allowing them to react. This reaction is exothermic (i.e., heat is released during the reaction) and is known as hydration. Here, water enters the cement crystals and forms a gel that is a hydrate, particularly calcium silicate hydrate (abbreviated C-S-H, with the average chemical formula $3\text{CaO} \cdot 2\text{SiO}_2 \cdot 3\text{H}_2\text{O}$). Partly because of the difference in volume between the reaction products and the original material, shrinkage (commonly known as drying shrinkage) accompanies the hydration reaction. In order to make sure that there is sufficient water available for the hydration reaction, the reaction is commonly conducted in a moisture chamber that has a high relative humidity (approaching 100%). During the first 24 h of the reaction, the originally fluid cement paste gradually becomes a rigid material that can thus be demolded if desired. This part of the hardening process is known as setting. However, the set material has not completed the hydration reaction, so the strength has not reached the desired high level. Therefore, setting is followed by curing for weeks (typically 28 days in laboratory studies). While curing, the material is exposed to moisture (as in a moisture chamber) for an extended period so as to allow the hydration reaction to approach completion.

Five types of Portland cement are standardized in the US by the American Society for Testing and Materials (ASTM). They are ordinary (Type I, which is the most commonly used type), modified (Type II), high-early-strength (Type III), low-heat (Type IV), and sulfate-resistant (Type V).

Effective use of discontinuous fibers in concrete requires dispersion of the fibers in the mix. The dispersion is particularly challenging when the fiber diameter is small, such as $10\text{ }\mu\text{m}$. The ease of dispersion increases with decreasing fiber length. The fiber dispersion is enhanced by using silica fume (a fine particulate) as an admixture at the level of, say, 15% by mass of cement. The silica fume is typically used along with a small amount (say 0.4% by mass of cement) of methylcellulose (a water-soluble polymer) to aid the dispersion of the fibers and the workability of the mix. Latex (typically a copolymer of styrene and butadiene in the form of a particulate dispersion, used typically at levels of 15–20% by mass of cement) is much less effective than silica fume at aiding fiber dispersion.

1.4 Composite Design Concepts

The filler can take the form of particles, discontinuous fibers or continuous fibers. The fibers can be oriented in one or more particular directions, or they can be randomly oriented. The combined use of different fillers is possible. For example, concrete is used in combination with sand and stones, which are different in particle size, in order to achieve a high total filler volume fraction. The fine particles fill the space between the large particles (Fig. 1.12), thus resulting in a total filler volume that cannot be achieved through the use of a single type of filler. In another example, carbon fiber and polymer fiber are used together in a polymer-matrix composite in order to achieve a balanced combination of high toughness (provided by the polymer fiber) and high modulus (provided by the carbon fiber). The two

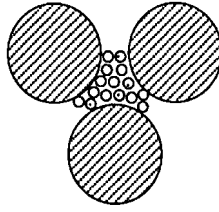


Figure 1.12. Packing of particles of two different sizes to attain a high density of packing

types of fibers can be in the same lamina or in different laminae of the composite. When they are in the same lamina, they can be in the same prepreg sheet or in the same woven fabric.

The matrix can be polymer, cement, metal, carbon, ceramic or a hybrid of multiple types of matrix materials. An example of a hybrid matrix is one consisting of both carbon and silicon carbide, as obtained, for example, by the pyrolysis of a precursor that is a mixture of a carbon precursor and a silicon carbide precursor. A hybrid matrix with carbon and silicon carbide is more oxidation resistant than a carbon matrix.

A special design of a hybrid matrix involves a spatial gradient in the proportion of the two components so that a certain property (e.g., the CTE) of the matrix varies along the composition gradient. Such a matrix is said to be functionally gradient. The functional gradient is made possible by a composition gradient. When the composite is used as an interface layer between article A and article B, such that A and B are different in CTE, it is desirable for the composite to be functionally gradient, so that its CTE in the part of it that is proximate to article A is close to that of A, and its CTE in the part of it that is proximate to article B is close to that of B. Such a CTE gradient helps to enhance the thermal fatigue resistance (i.e., the ability to withstand temperature cycling). An example is the coating of a carbon-carbon composite with silicon carbide for the purpose of oxidation protection, such that the interface layer has a composition gradient, with the side proximate to the carbon-carbon composite being rich in carbon and the side proximate to the silicon carbide being rich in silicon carbide.

In general, the longer the fiber, the greater its ability to reinforce a material. Thus, continuous fibers are dominantly used for polymer-matrix structural composites. However, discontinuous fibers are attractive for composite processing methods that require mixing of the filler and the matrix material to form a dispersion (or a slurry) that is introduced into the composite processing equipment. An example of such a processing method is injection molding, which involves the injection of the dispersion into a mold cavity that dictates the shape and size of the composite article. Injection molding is widely used to make polymer-matrix composite articles of complex shapes (such as telephone receivers). In order for the dispersion to conform to the intricate shape (fine details) of a mold for injection molding, it is advantageous for the filler to be small in size. Thus, nanofillers (nanoparticles or nanofibers) are attractive. Another example is concrete mixing, which results in a slurry that is poured into a mold (called a “form” in fieldwork) that dictates the shape and size of the concrete article (called a “struc-

ture" in fieldwork). Pouring into a mold is a process that is generally known as "casting."

In molding or casting, the fluidity (or workability) of the dispersion is important, particularly if the mold has an intricate shape. The fluidity can be enhanced by increasing the proportion of the liquid in the dispersion. However, the use of excessive liquid is often not desirable, as it may result in increased porosity in the resulting composite. In the case of a cement-matrix composite, the higher the water/cement ratio in the cement mix, the lower the strength of the resulting composite. An alternate method involves the use of a plasticizer, which is a chemical additive used in minor amounts. A plasticizer for cement is adsorbed onto the surfaces of the cement particles, thus causing a negative charge and hence electrostatic repulsion between cement particles. By separating the cement particles, water can reach more of the cement particles, thus causing the water present to be more effectively used for the hydration reaction. Thus, the plasticizer reduces the amount of water required, thus allowing the fluidity to be adequate without having to use a high water/cement ratio. This is why a plasticizer for cement is also known as a water-reducing agent. Examples of plasticizers for cement include lignosulfonates, sulfonated naphthalene condensate, and sulfonated melamine formaldehyde, which are organic polymers. Their long molecules wrap themselves around a cement particle and provide the particle with a negative charge.

Dispersion stability refers to the essential absence of a tendency for the filler in a dispersion to separate from the liquid part of the dispersion. In this context, stability should be distinguished from thermal stability. The liquid part is called the "vehicle" when it is based on a polymer or water. A vehicle based on a polymer or a polymer precursor (a resin) is often used to make a polymer-matrix composite. Vehicles based on water are used in cement-matrix composite fabrication. In metal-matrix composite fabrication, the liquid part is the molten metal.

Instability is usually associated with the filler units sticking to one another and then settling. The greater the difference in density between the filler and the liquid, the higher the tendency for instability. The stability is important for achieving a uniform distribution of the filler in the resulting composite. Matching the density of the filler to that of the liquid is a technique that is used to achieve stability. In general, the density of a liquid can be increased by dissolving an appropriate solid in the liquid. However, this method may not be able to give the required degree of stability, since the density of a liquid can be increased by only a limited degree.

Another method of improving the stability of a dispersion involves the use of a dispersant, which is a chemical additive that makes it more difficult for the filler units to aggregate. Filler aggregation is promoted by a low filler-filler interface energy and a high filler-liquid interface energy. By lowering the energy of the filler-liquid interface or increasing the energy of the filler-filler interface, the tendency for filler aggregation can be reduced. One method of increasing the energy of the filler-filler interface involves providing electrical charge to the filler units, thus resulting in electrostatic repulsion between the filler units. One method of decreasing the energy of the filler-liquid interface involves the use of a surfactant, which is a wetting agent that enhances the wettability of the liquid on the filler.

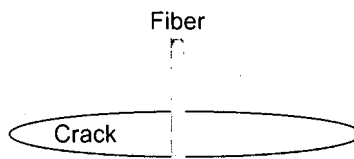


Figure 1.13. Fiber bridging a microcrack; microcracks tend to be present in brittle materials

Good wetting means that the liquid spreads across the surface of the filler. Poor wetting means that the liquid forms droplets on the surface of the filler. Another method of improving the wetting involves treating the surface of the filler by either coating the filler with a material that is more wettable by the liquid or applying chemical functionalization (i.e., the formation of functional groups on the surface of the filler that improve the wettability of the surface).

The ability of a fiber of a chosen length to reinforce a material increases with the strength of the bond between the reinforcement and the matrix. This bond depends on the microstructure and chemistry of the interface. For example, it can be enhanced by fiber surface treatment, such as a treatment that results in certain functional groups on the fiber surface (e.g., carboxyl and carbonyl groups on the surface of a carbon fiber). However, a bond that is too strong is disadvantageous to toughening when the toughening mechanism involves fiber pull-out (the pulling out of a fiber that bridges a microcrack as the microcrack opens, as illustrated in Fig. 1.13). Thus, for brittle-matrix composites, such as those with ceramic, carbon and cement matrices, the bond strength needs to be optimized.

The tailoring of composite interfaces is as important as the tailoring of composite components. Surface modification of a filler is commonly used for interface engineering. For example, this modification can involve the coating of the filler surface with molecules (e.g., a silane coupling agent) that have appropriate functional groups at their ends in order to provide some covalent linkage between the filler and the matrix across the interface. In other words, one end of the molecule attaches itself to the filler while the other end attaches itself to the matrix. There are many types of silane coupling agents that differ in the functional groups at the two ends of the molecule. For example, a molecule ending with a trichloro, trimethoxy or triethoxy functional group attaches to the surface of an oxide filler, whereas a molecule ending with an organofunctional functional group attaches to the polymer matrix. Because silane coupling agents are soluble in water, they are typically applied in the form of aqueous solutions. The use of a coupling agent is essential for glass fiber polymer-matrix composites. Otherwise, moisture attack causes debonding of the glass fiber from the polymer matrix.

1.5 Applications of Composite Materials

Due to their relatively low processing costs, polymer-matrix and cement-matrix composites are the most common types of composite. Polymer-matrix composites

with continuous fiber reinforcement are widely used for lightweight structures, such as airframes. Polymer-matrix composites with metal particles (e.g., silver particles) are used for electrical interconnections. Rubber-matrix composites reinforced with carbon black particles are used for automotive tires. Cement-matrix composites in the form of concrete are widely used for civil infrastructure. Metal-matrix, carbon-matrix and ceramic-matrix composites are less common, though they also have their particular markets.

Metal-matrix composites known as cermets (meaning ceramic-metal combinations) that contain a low volume fraction (e.g., 15%) of ceramic (e.g., tungsten carbide) particles are used in cutting tools such as drills. They are also used in resistors and other electronic components that need to withstand high temperatures. Metal-matrix composites containing ceramic (e.g., SiC) particles at a high volume fraction (e.g., 60 vol%) are used as heat sinks and housing for microelectronics due to their low CTE. A low CTE is needed due to the low CTE of the semiconductor (such as silicon). Metal-matrix composites containing graphite flakes as the filler are also used as self-lubricating piston cylinders for automobile engines due to the lubricity of graphite. Metal-matrix composites containing continuous carbon fibers are used as structural materials, though the reaction of carbon fiber with aluminum forms a brittle compound, Al_4C_3 , that lines the fiber-matrix interface. This reaction can be alleviated by coating the fiber with nickel or titanium diboride (TiB_2). However, this structural application faces tough competition from advanced metal alloys that are much less expensive.

Carbon-matrix composites (typically coated with silicon carbide or other ceramics in order to improve their oxidation resistance) are used for high-temperature, lightweight structures, such as the nose cones and leading edges of Space Shuttles and the nose cones of intercontinental ballistic missiles, although they suffer from the tendency of carbon to be oxidized in the presence of oxygen at temperatures above about 700°C. A particularly common type of carbon-matrix composites utilizes carbon fiber as the reinforcement, so that both reinforcement and matrix are carbon and the material is known as a carbon-carbon composite. The market for carbon-carbon composites is mainly related to aerospace: re-entry thermal protection, rocket nozzles and aircraft brakes. Other applications include furnace heating elements, molten materials transfer, spacecraft and aircraft components, and heat exchangers, air-breathing engine components, hypersonic vehicle airframe structures, space structures and prosthetic devices.

Biomedical applications encompass those that pertain to the diagnosis and treatment of conditions, diseases and disabilities, as well as the prevention of diseases and conditions. They include implants (e.g., hips, heart valves, skin and teeth), surgical and diagnostic devices, pacemakers (devices connected by electrical leads to the wall of the heart, enabling electrical control over the heartbeat), electrodes for collecting or sending electrical or optical signals for diagnosis or treatment, wheelchairs, devices for helping the disabled, exercise equipment, pharmaceutical packaging (for controlled release of the drug into the body, or for other purposes) and instrumentation for diagnosis and chemical analysis (such as equipment for analyzing blood and urine). Implants are particularly challenging, as they need to be made of materials that are biocompatible (compatible with fluids such as

blood), corrosion resistant, wear resistant, fatigue resistant, and that are able to maintain these properties over tens of years.

Carbon is a particularly biocompatible material (more so than gold), so carbon-carbon composites are used for implants. Composites with biocompatible polymer matrices are also used for implants. Electrically conducting polymer-matrix composites are used for electrodes for diagnostics. Composites with biodegradable polymer matrices are used for pharmaceuticals.

Materials for bone replacement or bone growth support need to have an elastic modulus similar to that of the bone. Tailoring of the modulus can be achieved through composite design, i.e., appropriate choice of the reinforcement and its volume fraction.

Ceramic-matrix composites are more attractive than carbon-matrix composites for high-temperature applications, due to the much lower tendency for ceramics to be oxidized. Examples of ceramic matrices include silicon carbide (SiC) and silicon nitride (Si_3N_4), which can withstand temperatures of up to around $1,700^\circ\text{C}$ in the presence of oxygen. Above $1,700^\circ\text{C}$, these ceramics can oxidize and become silicon dioxide (SiO_2). Ceramic-matrix composites with ceramic fiber reinforcement are known as ceramic-ceramic composites. It is preferable that the fiber and the matrix are the same in composition so that there is no CTE mismatch between them, for the sake of thermal fatigue resistance. One example of a ceramic-ceramic composite is a SiC - SiC composite. The ceramic reinforcement serves to toughen the composite. This is because of the tendency for microcracks to occur in the brittle ceramic matrix and the tendency for fiber bridging to occur across a microcrack (Fig. 1.13). The fiber pull-out that accompanies crack opening causes the absorption of energy and hence toughening. However, this mechanism of toughening requires that the bond between the fiber and the matrix is not too strong. Toughening is valuable, due to the inherent brittleness of ceramics. However, the technology of ceramic-matrix composites is not mature enough for implementation, due to both performance and cost issues.

Review Questions



1. Why is aluminum commonly used as the matrix of a metal-matrix composite?
2. Why is magnesium not commonly used as the matrix of a metal-matrix composite?
3. What are the main advantages of thermoplastic-matrix composites compared to thermoset-matrix composites?
4. Why is the pultrusion method of composite fabrication limited to unidirectional composites?
5. What is the main disadvantage of using woven fibers rather than nonwoven fibers as a reinforcement in a composite material?

6. Why is it difficult to use liquid metal infiltration to prepare metal-matrix composites with a low filler volume fraction?
7. What is the main disadvantage of the plasma spray method of metal-matrix composite fabrication?
8. Why is the stabilization step used in the fabrication of a carbon fiber from a pitch fiber?
9. Describe the CVI method of carbon-carbon composite fabrication.
10. What is the main advantage of having fillers of two different particle sizes in the same composite material?
11. Why is fiber bridging attractive for a brittle-matrix composite?

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Appendix: Test

Test Questions

This test consists of two parts covering a total of 6 pages. Part I consists of 16 multiple-choice questions. Part II consists of 17 questions in the conventional style.

Part I (32%)

Choose the best answer for each question.

1. (2%) In relation to structural vibration control, what is meant by active control?
 - a) Use of a viscoelastic material.
 - b) Use of a sensor.
 - c) Use of an actuator.
 - d) Combined use of a sensor and an actuator.
 - e) Energy dissipation.
2. (2%) Flexible graphite is a flexible sheet that is all graphite. The fabrication of flexible graphite involves the following procedure:
 - a) Intercalation.
 - b) Exfoliation.
 - c) Intercalation and exfoliation.
 - d) Compression.
 - e) Intercalation, exfoliation, and compression.
3. (2%) The fabrication of high-strength carbon fiber from pitch fiber involves the following procedure:
 - a) Carbonization.
 - b) Carbonization and graphitization.
 - c) Stabilization and carbonization.
 - d) Stabilization, carbonization, and graphitization.

4. (2%) What is the main advantage of pultrusion compared to compression molding for fabricating a continuous fiber polymer-matrix composite?
 - a) Higher fiber volume fraction.
 - b) Lower fiber volume fraction.
 - c) Long length of the resulting composite.
 - d) Composite has a constant cross-section.
 - e) Unidirectional configuration.
5. (2%) The rule of mixtures expression for the modulus of a unidirectional continuous fiber composite is:
 - a) $n = 0$ for the isostrain situation and $n = -1$ for the isostress situation.
 - b) $n = -1$ for the isostrain situation and $n = 1$ for the isostress situation.
 - c) $n = 1$ for the isostrain situation and $n = 0$ for the isostress situation.
 - d) $n = 0$ for the isostrain situation and $n = 1$ for the isostress situation.
 - e) $n = 1$ for the isostrain situation and $n = -1$ for the isostress situation.
6. (2%) Give an example of a glassy sealant that is used to improve the oxidation resistance of carbon-carbon composites.
 - a) HfC
 - b) B_2O_3
 - c) Ni
 - d) Si_3N_4
 - e) SiC
7. (2%) Describe the process of liquid metal infiltration involved in the creation of a SiC particle aluminum-matrix composite.
 - a) Mixing SiC particles with molten aluminum and subsequent casting.
 - b) Mixing SiC particles with molten aluminum and subsequent spraying.
 - c) A SiC particle preform is created and molten aluminum is forced into the preform.
 - d) Mixing SiC particles and aluminum particles and subsequent hot pressing above the melting temperature of aluminum.
 - e) Mixing SiC particles and aluminum particles and subsequent hot pressing below the melting temperature of aluminum.
8. (2%) Describe the coated filler method of powder metallurgy that is used to create a SiC particle copper-matrix composite.
 - a) Mixing SiC particles with molten copper and subsequent casting.
 - b) Mixing SiC particles with molten copper and subsequent spraying.
 - c) Mixing SiC particles and copper particles and subsequent hot pressing below the melting temperature of copper.

- d) Hot pressing copper-coated SiC particles below the melting temperature of copper.
- e) A SiC particle preform is created and molten copper is forced into the preform.
9. (2%) The rusting of iron in air involves the following anodic and cathodic reactions:
- $\text{Fe} \rightarrow \text{Fe}^{2+} + 2\text{e}^-$; $\text{O}_2 + 2\text{H}_2\text{O} + 4\text{e}^- \rightarrow 4\text{OH}^-$
 - $\text{Fe} \rightarrow \text{Fe}^{2+} + 2\text{e}^-$; $\text{O}_2 + 4\text{H}^+ + 4\text{e}^- \rightarrow 2\text{H}_2\text{O}$
 - $\text{Fe} \rightarrow \text{Fe}^{2+} + 2\text{e}^-$; $2\text{H}_2\text{O} + 2\text{e}^- \rightarrow \text{H}_2 \uparrow + 2\text{OH}^-$
 - $\text{Fe}^{2+} \rightarrow \text{Fe}^{3+} + \text{e}^-$; $\text{O}_2 + 2\text{H}_2\text{O} + 4\text{e}^- \rightarrow 4\text{OH}^-$
 - $\text{Fe}^{2+} \rightarrow \text{Fe}^{3+} + \text{e}^-$; $\text{O}_2 + 4\text{H}^+ + 4\text{e}^- \rightarrow 2\text{H}_2\text{O}$
10. (2%) Figure A.1 shows specimen configurations for measuring the shear bond strength between a ceramic and a metal (e.g., steel). In Fig. A.1a, the ceramic is bonded to only one side of the metal. In Fig. A.1b, the ceramic is bonded to both sides of the metal. The configuration in Fig. A.1b is advantageous compared to that in Fig. A.1a. Give the main reason.
- Larger joint interface area.
 - Larger amount of ceramic.
 - More brazing material.
 - The steel is better protected.
 - No bending moment during shear.
11. (2%) Why are both fine and coarse aggregates used in concrete?
- To attain a low total aggregate volume fraction.
 - To attain a high total aggregate volume fraction.
 - To decrease the fluidity of the mix.
 - To increase the fluidity of the mix.
 - To increase the rate of curing.

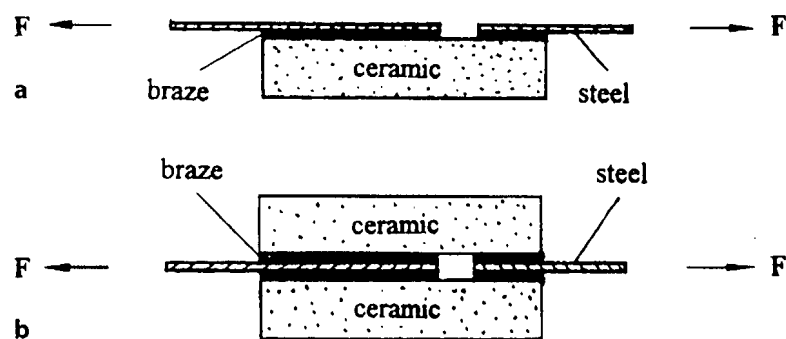


Figure A.1. Specimen configurations for measuring the shear bond strength between a ceramic and a metal (e.g., steel). **a** Ceramic is bonded to one side of the metal only. **b** Ceramic is bonded to both sides of the metal

12. (2%) What is the main advantage of using activated carbon fiber rather than activated carbon particles for water purification?
 - a) Faster water flow.
 - b) Greater surface area.
 - c) Larger pores.
 - d) Higher strength.
 - e) More flexibility.
13. (2%) Why are polymers typically advantageous compared to ceramics when used as electrically insulating layers in microelectronics?
 - a) Higher thermal conductivity.
 - b) Higher coefficient of thermal expansion.
 - c) Lower coefficient of thermal expansion.
 - d) Lower value of the relative dielectric constant.
 - e) Higher value of the relative dielectric constant.
14. (2%) A Bingham plastic refers to the following:
 - a) A material that exhibits Newtonian behavior.
 - b) A material that exhibits elastic behavior.
 - c) A material that exhibits shear yielding.
 - d) A material that exhibits shear thinning.
 - e) A material that exhibits shear thickening.
15. (2%) In relation to viscoelastic behavior, the quantity $\tan \delta$ refers to the following:
 - a) The ratio of the storage modulus to the loss modulus.
 - b) The ratio of the loss modulus to the storage modulus.
 - c) The magnitude of the shear modulus.
 - d) The product of the magnitude of the shear modulus and $\sin \delta$.
 - e) The product of the magnitude of the shear modulus and $\cos \delta$.
16. (2%) A remendable polymer is:
 - a) A thermoplastic polymer.
 - b) A thermosetting polymer.
 - c) A polymer that melts upon heating.
 - d) A polymer that decomposes upon heating.
 - e) A polymer for which the degree of crosslinking decreases reversibly upon heating.

Part II (68%)

1. (4%) Name two main advantages of high-modulus carbon fiber compared to high-strength carbon fiber.
2. (4%) A piezoresistive material exhibits a gage factor of 22. The gage factor is defined as the fractional change in resistance per unit strain. A piece of this material has a resistance of $46\ \Omega$. What is its resistance when the strain is 4.5%?
3. (4%) A unidirectional continuous fiber composite contains fibers of modulus 3.4 GPa. The fiber volume fraction is 57%. The modulus of the matrix is 0.12 GPa. Assuming that the bonding between the fiber and the matrix is perfect, calculate the modulus of the composite in the longitudinal direction (i.e., in the fiber direction).
4. (4%) In relation to carbon materials, the inhibition factor of a treated carbon is defined as the ratio of the oxidation rate of untreated carbon to the oxidation rate of the treated carbon. The oxidation rates are 9.9%/min for the untreated carbon and 2.8%/min for the treated carbon. Calculate the inhibition factor.
5. (4%) The rate r of a thermally activated process relates to the activation energy Q of the process by the equation

$$r = Ae^{-Q/RT}$$

where T is the temperature in K (not in $^{\circ}\text{C}$), R is the gas constant (a universal constant equal to $8.314\text{ J}/(\text{mol K})$) and A is just a proportionality constant. The rate at temperature T_1 is r_1 and the rate at temperature T_2 is r_2 . Derive an expression for Q in terms of T_1 , T_2 , r_1 and r_2 .

6. (4%) Using a graph, define the plastic viscosity and the apparent viscosity.
7. (4%)

$$\begin{aligned}\tau &= \eta, \\ \tau &= \tau_0 e^{i\omega t}, \\ \gamma &= \gamma_0 e^{i(\omega t - \delta)}\end{aligned}$$

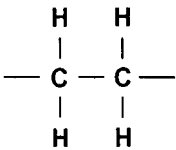
In the above equations that pertain to viscoelastic behavior, τ is the shear stress, γ is the shear strain, and η is the viscosity. Derive the equation

$$|\eta| = |G|/\omega,$$

where G is the shear modulus.

8. (4%) Using a graph of stress versus strain, explain the phenomenon of pseudoplasticity.
9. (4%) A brittle material is much stronger under compression than under tension. Explain the scientific origin of this phenomenon.

10. (4%) Polyvinyl chloride is stiffer than polyethylene. Explain the scientific origin of this observation. Note that the mer of polyethylene is



while the mer of polyvinyl chloride is the same as this except that one for the four hydrogen atoms has been replaced with a chlorine atom.

- 11. (4%) What are the main problems with the method of self-healing involving microcapsules of a monomer?
- 12. (4%) Describe an effective method of improving the vibration damping ability of a continuous carbon fiber polymer-matrix composite.
- 13. (4%) Why does the time spent below the melting temperature prior to bonding affect the quality of the bond for polyphenylene sulfide (PPS)?
- 14. (4%) Why is aluminum foil sufficiently temperature resistant that it can be used in cooking?
- 15. (4%) Why does corrosion tend to occur in a fastened metal joint?
- 16. (4%) Why does sand blasting help to improve the corrosion resistance of steel rebars?

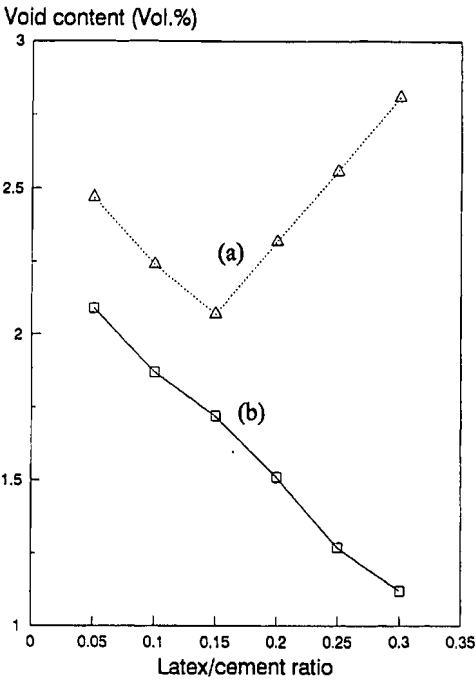


Figure A.2. Effect of the latex/cement ratio on the air void content of cement paste: **a** with 0.53 vol% carbon fibers; **b** with no fibers

67

17. (4%) Figure A.2 shows the effect of the latex/cement ratio on the air void content of cement paste. Curve (a) is for the case with 0.53 vol% carbon fibers. Curve (b) is for the case without fiber. Explain the features of curve (a) in terms of their scientific origin.

Test Solutions

Part I (32%)

The correct answers are:

1. d
2. e
3. c
4. c
5. e
6. b
7. c
8. d
9. a
10. e
11. b
12. a
13. d
14. c
15. b
16. e

Part II (68%)

1. Any two of the following answers are acceptable: higher modulus; higher thermal conductivity; higher electrical conductivity; higher oxidation resistance.
2. $[(R - 46)/46]/4.5\% = 22$
 $(R - 46)/46 = 0.99$
 $R - 46 = 45.54$
 $R = 91.5\Omega$.
3. Modulus = $(0.57) (3.4 \text{ GPa}) + (0.43) (0.12 \text{ GPa}) = 2.0 \text{ GPa}$.
4. Inhibition factor = $9.9\%/2.8\% = 3.5$.

5. $r_1 = A e^{-Q/RT_1}$

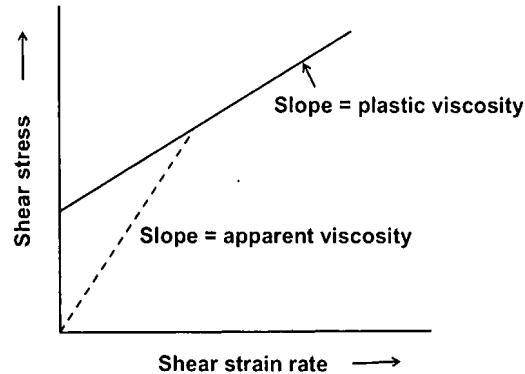
$r_2 = A e^{-Q/RT_2}$

$r_1/r_2 = e^{-Q/RT_1}/e^{-Q/RT_2} = e^{-Q/RT_1+Q/RT_2}$

$\ln(r_1/r_2) = -Q/RT_1 + Q/RT_2 = -(Q/R)[(1/T_1) - (1/T_2)]$

$Q = -R \ln(r_1/r_2)/[(1/T_1) - (1/T_2)]$.

6. The graph defining the plastic viscosity and the apparent viscosity should look like this:

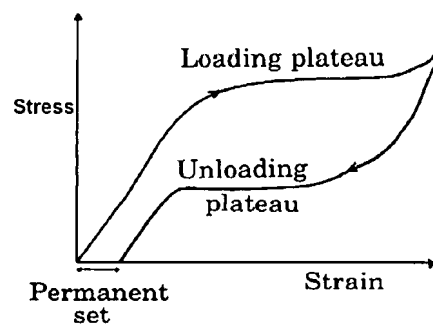


7. $\dot{\gamma} = \gamma_0 i \omega e^{i(\omega t - \delta)}$

$\eta = \tau_0 e^{i\omega t} / [\gamma_0 i \omega e^{i(\omega t - \delta)}] = -i(|G|/\omega) e^{i\delta}$

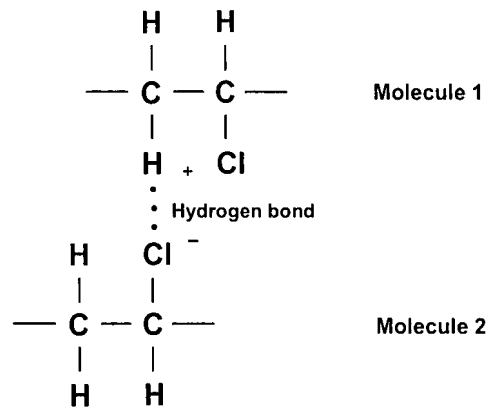
$|\eta| = |G|/\omega$.

8. A graph of stress versus strain that explains the phenomenon of pseudoplasticity should look like this:



9. The microcracks that tend to be present in a brittle material propagate much more under tension than under compression.

10. In PVC, there is hydrogen bonding (secondary bonding) between a hydrogen of one molecule and the chlorine of another molecule (the following diagram is not required):



11. Toxicity of the monomer; high cost of the catalyst.
12. Add either a viscoelastic material or a nanofiber material to the composite at the interlaminar interface (mentioning either material is sufficient).
13. PPS is a thermoplastic that has thermosetting character, so it cures to a limited degree as it spends time below the melting temperature prior to bonding.
14. It is covered with a nonporous protective layer of aluminum oxide.
15. The reduced availability of oxygen at the crevice in the fastened joint results in an oxygen concentration cell, meaning that the crevice region becomes the anode.
16. Sand blasting removes the impurities on the surface of the rebar, thus resulting in a more uniform surface composition.
17. The decrease in void content as the latex/cement ratio increases up to about 0.15 is due to the latex. The increase in void content as the latex/cement ratio increases beyond about 0.15 is due to the decreasing degree of fiber dispersion. (Fiber clumping increases the void content.)

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ANEXO 2

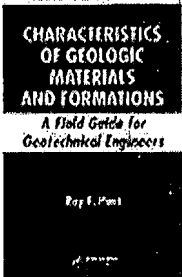


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1

Rock and Soil: Identification and Classification

1.1 Introduction

1.1.1 The Geologic Materials

Definitions

Precise definitions of the two general constituents, rock and soil, that are applicable to all cases are difficult to establish because of the very significant transition zone in which rock is changing to soil or in which a soil formation has acquired rock-like properties, or various other conditions. In general terms, the constituents may be defined as follows.

Rock

Material of the Earth's crust, composed of one or more minerals strongly bonded together that are so little altered by weathering that the fabric and the majority of the parent minerals are still present.

Soil

A naturally occurring mass of discrete particles or grains, at most lightly bonded together, occurring as a product of rock weathering either *in situ* or transported, with or without admixtures of organic constituents, in formations with no or only slight lithification.

Comments

The definitions given are geologic and not adequate for application to engineering problems in which the solution relates to hydraulic and mechanical properties as well as to certain other physical properties, such as hardness. For most practical engineering problems, it is more important to describe and classify the materials in terms of their physical conditions and properties than to attempt in every case to define the material as a soil or a rock.

The most important practical distinction between soil and rock in engineering works arises in excavations, since soil is normally much less costly to remove than rock, which may require blasting. For pay quantities, soils are usually defined as materials that can be removed by machine excavation, and other materials are defined as rock. This can be an ambiguous definition since the success of machine excavation depends on the size, strength, and condition of the equipment, as well as the effort applied by the operator.

In rock masses, the material factors of major significance in excavations, as well as in other engineering problems, are mineral hardness, the frequency and orientations of

fractures, and the degree of decomposition. Some geologic materials defined as rock, such as halite, many shales, and closely jointed masses, may be removable by machine, or may be more deformable under applied stress than are some materials defined as soils, such as glacial tills and cemented soils. Compounding the problem of definition is rock decomposition that, at a given location, can vary over short lateral and vertical distances, resulting in materials ranging from "hard" to "soft."

In the literature, rock can be found defined for engineering purposes as "intact specimens with a uniaxial compressive strength of the order of 100 psi (6.8 tsf, kg/cm²) or greater." For the above reasons, this definition is not applicable to rock masses in many instances.

General Classification

A broad classification which generally includes all geologic materials, together with a brief description, is given in Table 1.1.

1.1.2 Rock Groups and Classes

Geologic Bases

Based on their geologic aspects, rocks are grouped by origin as igneous, sedimentary, or metamorphic, and classified according to petrographic characteristics, which include their mineral content, texture, and fabric.

Engineering Bases

On an engineering basis, rock is often referred to as either intact or *in situ*.

Intact rock refers to a block or fragment of rock free of defects, in which its hydraulic and mechanical properties are controlled by the petrographic characteristics of the material, whether in the fresh or decomposed state. Classification is based on its uniaxial compressive strength and hardness.

TABLE 1.1

A Broad Classification of Geologic Materials

Category	Material	Description
Rock	Fresh rock	
	Intact	Unweathered rock free of fractures and other defects
Transition zone	Nonintact	Unweathered, but divided into blocks by fractures
	Decomposed rock	
	Intact	Weathered, the rock structure and fabric remain but mineral constituents are altered and the mass softened
Soil	Nonintact	Rock softened and altered by weathering and containing discontinuities (fractures)
	Residual soils	Most minerals changed by advanced decomposition; fabric remains or is not apparent; material friable
	Transported soils	The residual soils are removed and graded by the agents of transportation: wind, water, gravity, and glaciers. Degree of grading varies with transport mode
	Sedentary soils	Organic deposits develop <i>in situ</i> from decomposition of vegetation
Either	Duricrusts	Deposits from evaporation of groundwater: caliche, laterite, ferrocrete, silcrete

In situ rock refers to the rock mass that normally contains defects, such as fractures or cavities, which separate the mass into blocks of intact rock and control the hydraulic and mechanical properties. Classification is based on rock quality, with the mass generally termed as competent or incompetent. Various practitioners have presented systems for describing incompetent rock, which can contain a wide range of characteristics, but a universally accepted nomenclature has not been established.

1.1.3 Soil Groups and Classes

Geologic Bases

Geologically, soils are grouped or classified on a number of bases, as follows:

- Origin: residual, colluvial, alluvial, aeolian, glacial, and sedentary
- Mode of occurrence: floodplain, estuaries, marine, moraine, etc.
- Texture: particle size and gradation
- Pedology: climate and morphology

Engineering Bases

Classes

Soils are classified on an engineering basis by gradation, plasticity, and organic content, and described generally as cohesionless or cohesive, granular, or nongranular.

Groups

Soils are grouped by their engineering characteristics as strong or weak, sensitive or insensitive, compressible or incompressible, swelling (expansive) or nonswelling, pervious or impervious; or grouped by physical phenomena as erodible, frost-susceptible, or metastable (collapsible or liquefiable, with the structure becoming unstable under certain environmental changes).

Soils are also grouped generally as gravel, sand, silt, clay, organics, and mixtures.

1.2 Rocks

1.2.1 The Three Groups

Igneous

Igneous rocks are formed by the crystallization of masses of molten rock originating from below the Earth's surface.

Sedimentary

Sedimentary rocks are formed from sediments that have sometimes been transported and deposited as chemical precipitates, or from the remains of plants and animals, which have been lithified under the tremendous heat and pressure of overlying sediments or by chemical reactions.

Metamorphic

Metamorphic rocks are formed from other rocks by the enormous shearing stresses of orogenic processes that cause plastic flow, in combination with heat and water, or by the

heat of molten rock injected into adjoining rock, which causes chemical changes and produces new minerals.

1.2.2 Petrographic Identification

Significance

Rocks are described and classified by their petrographic characteristics of mineral content, texture, and fabric.

A knowledge of the mineral constituents of a given rock type is also useful in predicting the engineering characteristics of the residue from chemical decomposition in a particular climatic environment. Residual soils are commonly clayey materials, and the "activity" of the formation is very much related to the original rock minerals.

Rock Composition

Minerals

Rock minerals are commonly formed of two or more elements, although some rocks consist of only one element, such as carbon, sulfur, or a metal.

Elements

Oxygen, silicon, aluminum, iron, calcium, sodium, potassium, and magnesium comprise 98% of the Earth's crust. Of these, oxygen and silicon represent 75% of the elements. These elements combine to form the basic rock minerals.

Groups

The mineral groups are silicates, oxides, hydrous silicates, carbonates, and sulfates. Silicates and oxides are the most important. The groups, mineral constituents, and chemical compositions are summarized in Table 1.2. Chemical composition is particularly important as it relates to the characteristics of materials resulting from chemical weathering and decomposition.

Texture

Texture refers to the size of grains or discrete particles in a specimen and is generally classified as given in Table 1.3.

Fabric

Fabric refers to grain orientation, which can be described in geologic or in engineering terminology.

Geologic Terminology

- Equigranular: grains essentially of equal size
- Porphyritic: mixed coarse and fine grains
- Amorphous: without definite crystalline form
- Platy: schistose or foliate

Engineering Terminology

- Isotropic: the mineral grains have a random orientation and the mechanical properties are the same in all directions.

TABLE 1.2
Common Rock-Forming Minerals and Their Chemical Composition

Group	Mineral	Variety	Chemical Composition	Comments
Silicates	Feldspars	Orthoclase	$KAlSi_3O_8$	Very abundant
		Plagioclase	$NaAl_2Si_2O_8$	
			$CaAl_2Si_2O_8$ } Variable	
	Micas	Muscovite (white mica)	$KAl_2(Si_3Al)O_{10}(OH)_2$	Very abundant
		Biotite (dark mica)	$K_2(MgFe)_6(SiAl)_8O_{20}(OH)_4$	Ferromagnesian mineral ^a
	Amphiboles	Hornblende	Na, Ca, Mg, Fe, Al silicate	Ferromagnesian mineral ^a
Hydrous silicates	Pyroxenes	Augite	Ca, Mg, Fe, Al silicate	Ferromagnesian mineral ^a
	Olivine		$(MgFe)_2SiO_4$	Ferromagnesian mineral ^a
	Kaolinite		$Al_2Si_2O_5(OH)_4$	Secondary origin ^b
	Quartz		SiO_2	Very abundant
Oxides	Aluminum		Al_2O_3	
	Iron oxides	Hematite	Fe_2O_3	
		Limonite	$2Fe_2O_3 \cdot 3H_2O$	
		Magnetite	Fe_3O_4	
Carbonates	Calcite		$CaCO_3$	Very abundant
	Dolomite		$CaMg(CO_3)_2$	
Sulfates	Gypsum		$CaSO_4 \cdot 2(H_2O)$	
	Anhydrite		$CaSO_4$	

^a Ferromagnesian minerals and the iron oxides stain rock and soils orange and red colors upon chemical decomposition.
^b Hydrous silicates form from previously existing minerals by chemical weathering and include the clay minerals (such as kaolinite and the chlorites), serpentine, talc, and zeolites.
^c Magnetite occurs in many igneous rocks and provides them with magnetic properties even though often distributed as very small grains.

TABLE 1.3
Textural Classification of Mineral Grains

Class	Size Range (mm)	Recognition
Very coarse-grained	>2.0	Grains measurable
Coarse-grained	$0.6-2.0$	Clearly visible to eye
Medium-grained	$0.2-0.6$	Clearly visible with hand lens
Fine-grained	$0.06-0.2$	Just visible with hand lens
Very fine-grained	<0.06	Not distinguishable with hand lens

- Anisotropic: the fabric has planar or linear elements from mineral cleavage, foliations, or schistose, and the properties vary with the fabric orientation.

Mineral Identification Factors

Crystal Form

Distinct crystal form is encountered only occasionally in rock masses. Some common minerals with distinct forms are garnet, quartz, calcite, and magnetite.

Color

Clear or light-colored minerals include quartz, calcite, and feldspar. Dark green, dark brown, or black minerals contain iron as the predominant component.

ANEXO 1

ANEXO 2

ANEXO 3



The New Encyclopædia Britannica

Volume 10

MICROPÆDIA

Ready Reference

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short imprisonment, was allowed to cross over to France. He was greatly trusted by Charles II, whose defeat at Worcester and subsequent wanderings he shared, and during this king's exile he was one of his principal advisers, being created earl of Rochester in 1652. In the interests of Charles he engaged in several diplomatic missions on the continent; and in March 1655 he was in England, where he led a feeble attempt at a rising on Marston Moor, near York. On its failure he fled the country.

• **Rochester, John Wilmot, 2nd earl of** (b. April 10, 1647, Ditchley Manor House, Oxfordshire, Eng.—d. July 26, 1680, Woodstock, Eng.), court wit and poet who helped establish English satiric poetry.

Wilmot succeeded his father to the earldom in 1658, and he received his M.A. at Oxford in 1661. Charles II, probably out of gratitude to the 1st earl, who had helped him to escape after the Battle of Worcester (1651), gave the young earl an annual pension and appointed Sir Andrew Balfour, a Scottish physician, as his tutor. They travelled on the Continent for three years until 1664.

On his return, as a leader of the court wits, Rochester became known as one of the wildest debauchees at the Restoration court, the hero of numerous escapades, and the lover of various mistresses. Among them was the actress Elizabeth Barry, whom he is said to have trained for the stage, and an heiress, Elizabeth Malet. He volunteered for the navy and served with distinction in the war against the Dutch (1665-67). In 1667 he married Elizabeth Malet and was appointed a gentleman of the bedchamber to the king. In 1673 John Dryden dedicated to Rochester his comedy *Marriage à la Mode* in complimentary terms, acknowledging his help in writing it.

Rochester is generally considered to be the most considerable poet and the most learned among the Restoration wits. A few of his love songs have passionate intensity. He is also one of the most original and powerful of English satirists. His "History of Insuper" (1676) is a devastating attack on the government of Charles II, and his "Maim'd Debauchee" has been described as "a masterpiece of heroic irony." *A Satyr Against Mankind* (1675) anticipates Swift in its scathing denunciation of rationalism and optimism and in the contrast it draws between human perfidy and folly and the instinctive wisdom of the animal world.

In 1675 Rochester was appointed ranger of Woodstock Forest, where much of his later poetry was written. His health was declining, and his thoughts were turning to serious matters. His correspondence (dated 1679-80) with the Deist Charles Blount shows a keen interest in philosophy and religion, further

stimulated by his friendship with Gilbert Burnet, later bishop of Salisbury. Burnet recorded their religious discussions in *Some Passages of the Life and Death of John, Earl of Rochester* (1680). In 1680 he became seriously ill and experienced a religious conversion, followed by a recantation of his past; he ordered "all his profane and lewd writings" burned.

His single dramatic work, the posthumous *Valentinian* (1685), an attempt to rehandle a tragedy of Fletcher, contains two of his finest lyrics. His letters to his wife and to his friend Henry Savile are among the best of the period and show an admirable mastery of easy, colloquial prose.

A biography by Graham Greene, *Lord Rochester's Monkey*, was published in 1974.

• **Rochester, Lawrence Hyde, 1st earl of**, also called (1681-82) **VISCOUNT HYDE OF KENILWORTH** (b. March 1642—d. May 2, 1711, London), influential English statesman who served under Charles II, James II, William III, and Queen Anne.



Lawrence Hyde, 1st Earl of Rochester, detail of an oil painting after Willem Wissing, c. 1685-87, in the National Portrait Gallery, London
By courtesy of the National Portrait Gallery, London

The second son of the renowned statesman and historian Edward Hyde, 1st earl of Clarendon, he entered Parliament in 1660 and was master of the robes from 1662 to 1675. In 1679 he was made first lord of the Treasury and a privy councillor.

Hyde helped defeat Whig efforts (1678-81) to exclude from the succession the Catholic James, duke of York (later King James II), whose first wife had been Hyde's sister. In 1681 Hyde negotiated with the French a secret subsidy that enabled Charles II to remain independent of the Whig-dominated Parliament. He was created a viscount in 1681 and an earl in 1682.

For reasons that are not clear, Rochester was removed from the Treasury in 1684 but was given the lord lieutenantcy of Ireland and the relatively powerless position of lord president of the Council. Under James II, Rochester was lord treasurer from 1685 to 1687 but fell from power as James endeavoured to implement his pro-Catholic policies.

Though he was appointed to the Privy Council in 1692, he failed to gain the favour of King William III until late in the reign, when he was lord lieutenant of Ireland (1701-03). In 1702-04 he published his father's great work, *History of the Rebellion and Civil Wars in England*. Upon the accession of Queen Anne, he became a leader of the Tory High Church Anglican party, and in 1710-11 he was again president of the Council.

Rochester upon Medway, formerly **MEDWAY**, district (borough), county of Kent, England, with an area of 62 sq mi (160 sq km). The district is named for, and lies around the mouth of, the River Medway where it flows

into the estuary of the Thames. It comprises the cathedral city of Rochester, the town and naval dockyard of Chatham (centres that, together with nearby Gillingham, are sometimes referred to as the "Medway Towns"), and a section of the marshes and flats along the Thames. A detached portion of the latter, the Isle of Grain, has been dyked and reclaimed and is now the site of a large oil refinery. The lower Medway is an industrialized area. Since the 15th century Chatham has had one of Britain's major naval dockyards, and, besides this and the oil refinery at Grain, there are numerous engineering plants lining the river frontage of the Medway. Pop. (1983 est.) 146,200.

Rochford, district, county of Essex, England, bordered on the north by the unbridged River Crouch and on the south by the Southend-on-Sea urban area on the Thames Estuary. The River Roach (formerly Roch) flows through the centre of the district and into the North-Sea among the islands, marshes, and mud flats that form the easternmost part of the district. The coast is much used for sailing and recreation, while the inland parts of the district lie within the commuter belt of London. The area of Rochford is 65 sq mi (169 sq km). Pop. (1983 est.) 74,800.

rock, in geology, naturally occurring and coherent aggregate of one or more minerals. Such aggregates constitute the basic units of which the solid Earth is comprised and typically form recognizable and mappable volumes. Rocks are commonly divided into three major classes according to the processes that resulted in their formation. These classes are (1) igneous rocks, which have solidified from molten material called magma; (2) sedimentary rocks, those consisting of fragments derived from preexisting rocks or of materials precipitated from solutions; and (3) metamorphic rocks, which have been derived from either igneous or sedimentary rocks under conditions that caused changes in mineralogical composition, texture, and internal structure. These three classes, in turn, are subdivided into numerous groups and types on the basis of various factors, the most important of which are chemical, mineralogical, and textural attributes.

For the composition, physical properties, formation, and distribution of the major classes of rocks and their subgroupings, see **MACROPAEDIA: Minerals and Rocks**; for petrology, the scientific study of rocks, see **Earth Sciences, The**.

For a description of the place of rocks in the circle of learning and for a list of both **MACROPAEDIA** and **MICROPAEDIA** articles on the subject, see **PROPAEDIA: Part Two, Division I**.

rock, also called **ROCK AND ROLL**, in music, a style that arose in the United States in the mid-1950s and through both the assimilation and evolution of other styles and the gradual broadening of the name itself came to be the dominant form of popular music, spreading as well to Europe and other parts of the world. Although rock music utilized a wide variety of instruments, the basic elements were one or several vocalists, an assortment of heavily amplified electric guitars (including bass, rhythm, and lead), and drums. Among the other instruments that could be used were the electric piano, electric organ, and music synthesizer, along with horns, woodwinds, and minor percussion instruments. In its early stages, it was a fundamentally simple style, relying on heavy, dance-oriented rhythms, common melodies and harmonies, and lyrics sympathetic to its audience's concerns—youth love, the stresses of adolescence, and automobiles. In its maturity rock tended to exhibit more complexity, and some of its most talented exponents attracted the notice of serious critics of music.



John Wilmot, 2nd earl of Rochester, detail of an oil painting attributed to J. Huyemans, in the National Portrait Gallery, London
By courtesy of the National Portrait Gallery, London

chokin and the Poles agreed upon the Truce of Andrusovo (January 1667), which placed the Ukraine east of the Dniepr River, as well as the city and region of Kiev, under Russian control. Following his diplomatic success, Ordyn-Nashchokin was promoted to the rank of boyar, appointed minister of foreign affairs, and also made head of several other departments, including the Ukrainian department (Malorossiysky Prkaz).

Assuming the role of Alexis' chief adviser, Ordyn-Nashchokin not only involved himself in foreign policy but also, foreshadowing Peter I the Great (reigned 1682-1725), advocated using western Europe as a model for Russian development. He promulgated a trade ordinance protecting Russian merchants from both foreign competition and native bureaucrats (1667), advanced the prospect of trade with Persia and Central Asia, and promoted interest in the shipbuilding industry.

Nevertheless, as Ordyn-Nashchokin continued to view expansion into the Baltic region as the major objective of Russian foreign policy and to favour a policy of friendship with Poland, which precluded an attempt to seize control of the remainder of the Ukraine, he came into conflict with the tsar, who dismissed him from the Ukrainian department in 1669 and from the Foreign Office in 1671. In February 1672 Ordyn-Nashchokin took monastic vows and subsequently only briefly resumed his lay career to conduct negotiations with the Poles in 1679.

Ordzhonikidze (Russia): see Vladikavkaz.

Ordzhonikidze, Grigory Konstantinovich, byname **SERGO** (b. Oct. 12 [Oct. 24, New Style], 1886, Gorsha, Russia—d. Feb. 18, 1937, Moscow), communist leader who played a major role in bringing Georgia under Soviet rule and in industrializing the Soviet Union.

Having joined the Bolshevik faction of the Russian Social Democratic Workers' Party in 1903, Ordzhonikidze was active in the revolutionary movement, becoming a member of the party's central committee (1912). In April 1912, however, he was arrested for the third time. He resumed his activities only after the imperial Russian government had been overthrown (February 1917) and he had returned from exile in Siberia.

Ordzhonikidze then became a member of both the executive committee of the Petrograd Soviet (the revolutionary council of workers and soldiers that rivaled the authority of the provisional government) and of the Bolshevik Party committee. After the Bolsheviks seized power (October 1917), he became commissar extraordinary for the Ukraine area (1918), a member of his party's Central Committee (1921), and chairman of the Central Committee's Caucasian bureau (1921). Despite Lenin's objections to his brutal methods (which were approved by Joseph Stalin) and the opposition of the local communist organizations, Ordzhonikidze helped the Red Army conquer Georgia, then merged Georgia with Armenia and Azerbaijan to create the Transcaucasian Federal Republic, which in turn was compelled to join Russia, Belorussia, and the Ukraine to form the Soviet Union (December 1922).

During the intraparty power struggles of the mid-1920s, Ordzhonikidze generally supported Stalin. Although he incurred the hostility of Lavrenty Beria, who as chief of the secret police in Transcaucasia forced him to transfer to the northern Caucasus (1926), Ordzhonikidze nevertheless advanced in 1926 to become a candidate member of the Central Committee's Politburo, chairman of the party's central control commission, which was responsible for eliminating dissension among party members, and commissar for worker-peasant inspection. In 1930 he became a full member of the Politburo.

Having helped organize the development of

Soviet industry during the First Five-Year Plan (1928-32), Ordzhonikidze became commissar for heavy industry in 1932. During the mid-1930s he opposed Stalin's industrial policy and displayed his disapproval of Stalin's terroristic rule. Although Ordzhonikidze's sudden death in 1937 was officially attributed to natural causes, Nikita Khrushchev later charged (1956) that Stalin had driven Ordzhonikidze to commit suicide.

ore, aggregate of economically important minerals sufficiently rich to enable separation of the minerals at a profit.

A brief treatment of ore deposits follows. For full treatment, see *MACROPAEDIA: Industries, Extraction and Processing*.

Originally the term ore was applied only to metallic minerals, but its definition has subsequently been broadened to include such non-metallic substances as sulfur, calcium fluoride (fluorite), and barium sulfate (barite) that have been deposited in the host rock after its formation. Although more than 2,800 minerals have been identified, only about 100 are considered ore minerals—e.g., chalcopyrite, bornite, and chalcocite, which are the principal sources of copper, and sphalerite and galena, the principal sources, respectively, of zinc and lead. Copper, molybdenum, and gold are commonly found in disseminated deposits, i.e., scattered more or less uniformly through a large volume of rock or gangue (unwanted minerals). Copper, lead, and zinc are frequently found in massive sulfide deposits, which have a high ratio of ore minerals to gangue. Many such deposits are believed to have been formed by precipitation from volcanic exhalations on the seafloor or by metasomatic replacement (a process of simultaneous solution and deposition). Characteristic massive sulfide deposits can be found at Broken Hill in New South Wales, Australia (lead and zinc), and at the Sullivan deposit in Ontario, Canada (copper). Some ore minerals, such as the lead-zinc ores of the Mississippi River valley, are confined within a single stratum or group of sedimentary strata.

ore dressing, mechanical treatment of crude ores to separate out the valuable minerals. Formerly applied only to ores of precious metals such as gold and silver, ore-dressing processes came to be used to recover other metals and nonmetallic minerals and such materials as graphite, sulfur, mica, feldspar, asbestos, and fluorspar.

The primary operations are crushing, comminution, or grinding; and beneficiation, or concentration. Other important operations are sizing and classification, settling and filtering, drying and heat treating, and agglomerating, or pelletizing.

Comminution is customarily carried out by primary and secondary crushers, followed by fine grinders consisting of rotating cylindrical mills that contain steel balls (ball mill), rods (rod mill), flint pebbles (pebble mill), or lumps of the rock or ore itself. Mills may be operated either wet or dry.

In beneficiation, the simplest method of concentration is handpicking to remove either the rich material or the waste material from a moving table or conveyor belt. Flotation may be used to separate materials of different buoyancy. Gravimetric methods of concentration utilize the differences in the densities of the wanted and unwanted materials to effect their separation. Jigging is a method of concentrating heavy minerals by using an open box, the jig, with a perforated bottom through which pulsating water currents are forced. Crushed ore fed into the top is stratified by the action of the water currents, the heavier minerals settling to the bottom. Another method uses shaking tables, also called sand or slime tables, depending on the particulate size of the material they treat. They are inclined slightly from the horizontal and operated with a reciprocating

action that moves the heavy minerals to the end of the table, while water washes the lighter minerals over the side into a container below. Pneumatic jigs and tables operate on the same principles but are applied to mineral ores of a light, fluffy nature, such as asbestos. Magnetic properties may be the basis for separation of minerals containing iron; minerals having electrostatic properties may be separated by an electric field. Ordinary screening devices may be used on many ores in which the valuable mineral is finer or coarser than the gangue, or ore residue.

Hydraulic classification depends on the different rates of settling of mixed sizes of grains in water. In air classification, air is the medium instead of water. Centrifugal classifiers use centrifugal force to separate coarse and fine particles.

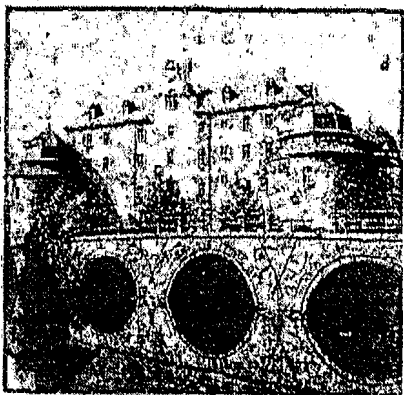
Settling and filtering remove water from the mixture. Drying is usually carried out in a rotary kiln, an inclined rotating cylinder with a burner at one end. Heat treating or roasting may be used to change the form or characteristic of a mineral in the ore to make it easier to concentrate.

Agglomeration (variously called sintering, pelletizing, and briquetting), the formation into tough lumps of material that cannot be smelted as fine particles, may be carried out by fusing the material. A binder such as starch, molasses, or bentonite may be mixed with the fine ore and the mixture heated or subjected to pressure.

Ore Mountains (Czechoslovakia-Germany): see Erzgebirge.

Örebro, län (county) of south-central Sweden, extending from the Bergslagen mining region in the north to Lake Hjälmars in the east and Lake Vättern in the south. Its land area of 3,289 square miles (8,519 square km) comprises the traditional *landskap* (province) of Närke and parts of those of Västmanland and Värmland. The län has a number of lakes and is drained by several rivers that flow south and east. Iron, zinc, and copper deposits are exploited; other important industries include lumbering, papermaking, steel making, and metal smelting. The capital and major city is Örebro, at the western edge of Lake Hjälmars. Pop. (1990 est.) 271,523.

Örebro, town and capital of Örebro län (county), south-central Sweden, on the Svart River at its entrance into Lake Hjälmars. One of Sweden's oldest towns, it was already a commercial centre in the 13th century and played a prominent part in Swedish history. It was the residence of Engelbrekt Engelbrektsson, leader of a rebellion against Denmark in 1434, and the birthplace of Olaus and Lauren-



Castle on an island in the Svart River, Örebro, Sweden
Reier