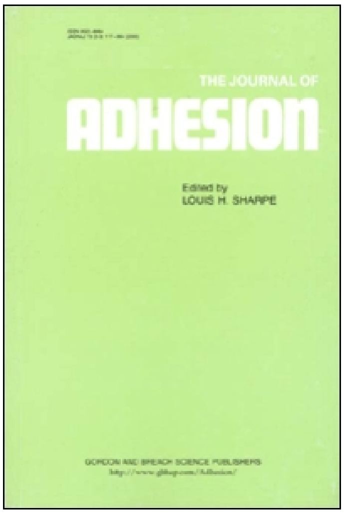

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Interfaces in Metal-Matrix Composites†

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Although metal-matrix composites are geometrically similar to non-metal-matrix composites, the components are sufficiently dissimilar that the two classes have different applications and problems. Metal-matrix composites are generally used at elevated temperatures, so system stability is an important consideration. Interfaces in these composites serve as the bonding surfaces and also as the site of component interaction. The component interactions may take any of several physical or chemical forms, depending on the specific composite under consideration. This paper deals with the nature of the constituents, kinds of interfaces, and the relation between interfacial considerations and composite utilization for both fabricated (solid- or liquid-matrix phase) and eutectic metal-matrix composites. The relation of metal- to nonmetal-matrix systems is emphasized throughout.

INTRODUCTION

In the study of composite materials, some paths of investigation are properly viewed as applicable to both metal- and nonmetal-matrix systems. Elasticity of inhomogeneous media and fastening methods are two such areas. However, the nature of the constituents or the use of the composite may affect the appropriateness or applicability of certain research. For example, the response of metal-matrix composites to elevated-temperature exposure is fundamentally different from that of nonmetal-matrix composites, as the physical changes of the two matrix materials are different. Moreover, some studies need not be performed on one type but are a requisite to the use of

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the other: reinforcement coarsening is never a problem in plastic-matrix composites, but it must be studied for directionally solidified eutectic composites; understanding the response of plastic-matrix composites to moisture-containing environments is vitally important to their use, but that consideration is secondary for most metal-matrix composites.

A principal theme of this conference is to promote interaction of scientists approaching their composite systems in different ways. In order to learn from the others, each group must understand the problems faced by its counterparts working in different types of systems, why certain approaches are useful and the physical situations which necessitate those approaches. This paper presents a review of current concepts of and problems associated with interfaces in metal-matrix composites with particular emphasis on similarities in and differences between metal- and nonmetal-matrix systems. The topics covered are the nature of the constituents, bonding, kinds of interfaces, how interfacial considerations influence usage, and experimental methods. Metal-matrix composites may contain several types of interfaces, but the present paper deals only with those between reinforcement and matrix.

CONSTITUENT PROPERTIES

While metals and nonmetals (plastics) have certain similarities, their differences define the basis for each's selection for composite applications. The most important feature of metals is their strong nondirectional metallic (free electron) bonding.[1] Metallic atoms are arranged periodically in crystals, in contrast to those of nonmetals. The crystallographic orientation determines the measured values of many properties,[2] for example modulus, surface energy, and, to some extent, strength. Although generally the modulus, flow stress, strength, hardness, and erosion resistance of metals are high, discontinuities and flaws on a fine scale can greatly influence their deformation and ductility.[3] One type of discontinuity, the dislocation, has been used to explain many aspects of the flow and fracture of metals.[4] The good elevated temperature strength and ductility of metals, coupled with their high density, means that their use is technically and economically favorable in structures exposed to high temperatures. At high temperatures self- or second-component diffusion can occur rapidly, often resulting in the formation of solid solutions or compounds in multicomponent systems.[5] Related phase transformations may then be observed, some of which can be used to improve the properties of metals.[6] Finally, the bonding of metals imparts to them nonmechanical properties of interest, such as good electrical and thermal conductivity. A second major difference between metals and nonmetals is their response to external environments.[7] Metals are attacked by different gaseous and liquid chemical agents than are organics or ceramics. Also, their temperature dependence of properties and stability at elevated temperatures are different. Response to these external influences is a prime consideration in choosing composite matrix materials.[8]

BONDING

In order to form a composite, the components must be combined at an interface. The direct contact bonding of two materials is a two-step process.[9] First, the parts must be brought into close proximity so that the contact area between them is maximized. Then a bond between atoms on either side of the bond line must be effected. The two processes can be treated separately.

Production of maximum contact

With nonmetal matrices the principal requirement for maximum contact is that all pores at the interface be removed. The bonding process is schematically shown in Figure 1. If the polymer "wets" the surface—that is, spreads on it—then most voids are immediately eliminated.[10] Because of the relatively low polymer flow stress, pressure during formation causes interface asperities to be flattened and some pores to be eliminated. Remaining pores do, however, decrease the interfacial contact, and pores in the interface influence composite deformation.

Metal-matrix composites are produced either by liquid- or solid-state-matrix techniques. With liquid-phase methods the first requirement is that the liquid matrix wet the substrate, exactly as with an organic matrix. As with that case, wetting leads to the elimination of voids or pores at the interface. The theory of the effect of surface active additions to metals has been developed[11] and serves as a guide to methods of reducing the melt surface energy, thereby improving its spreading properties.[12] When solid-state press- or roll-bonding techniques are used, the high pressure flattens surface asperities to promote close contact between the mated parts.[13] Remaining pores should be removed if high bond strength is desired, but the available mechanisms for the process are different than in the organic-matrix case. Individual units of the pore—vacancies—may leave the main pore and diffuse along or away from the interface to a free surface if such processes can be made thermodynamically favorable. If, however, the pore contains components insoluble in the metal, they may be permanently

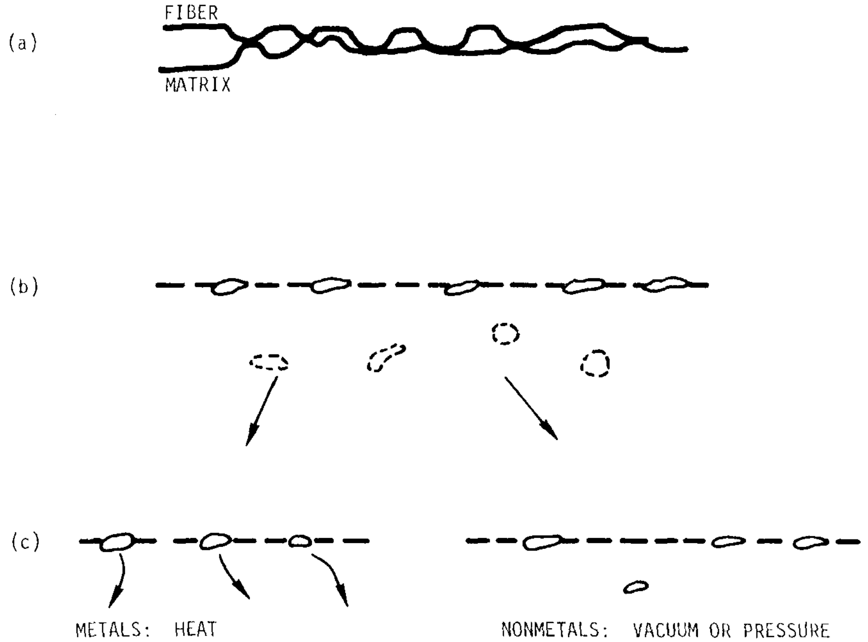

FIGURE 1 Schematic drawing of the bonding process. First contact is made and asperities reduced in size. Finally, interface pores may either float away (nonmetals) or break into easily diffusing voids (metals).

retained at the bond line. Solid-state bonding procedures are usually accomplished at elevated temperatures in order to reduce the metal's flow stress, facilitating the flattening of asperities and promoting vacancy diffusion. In some techniques[14] (e.g., electrodeposition) the constituents are bonded at low temperatures and then sintered. In both metal- and nonmetal-matrix composites voids decrease effective shear and normal bond strength and also act as stress concentrators.

In addition to decreasing interface porosity, metal-matrix bonding processes should also reduce bondline contaminant content, thus allowing clean metal surfaces to contact each other.[13] The bonding of aluminum is more difficult than that of titanium, for example, since aluminum has an insoluble protective adherent oxide while titanium readily dissolves its surface oxide at bonding temperatures.[15] The act of mechanical pressing or rubbing of the surfaces cracks the oxide film. Other methods of removing the contaminant include cleaning, fluxing, or creating a low-melting eutectic in the near-surface region.[13] Because the oxide contaminants generally float on liquid metals, composites produced from metallic melts in proper atmospheres are not troubled by oxide problems to the extent that solid-state-bonded composites are. In practical cases contamination from other sources (such as oil, dirt, tooling, or inert gases) can deter the attainment of full contact, but these problems are usually eliminated by careful attention to process control.

Chemical bonding

After contact is achieved, bonding may occur. The bonding in organic-matrix composites is usually either the dipole or the covalent type,[16] and the concept of an interphase region is often useful.[17] Covalent bonds generally occur between atoms of the matrix and fiber proper or with coupling agents such as silanes.[18] However, layers of water often reside on the surfaces even following cleaning,[19] so the actual bonds in that case are with water molecules. Because moisture diffuses along the interfaces rapidly, this situation is not necessarily damaging, since the bonds produced during fabrication are then the same as those found in normal service. Methods[20] to analyze the sensitivity of bonds to moisture or other environments have been developed for plastic-matrix composites.

Ideally,† metallic bonding at interfaces in composites is a result of the existence of an equilibrium between attraction and repulsion of metal atoms, as described by the familiar Lennard-Jones potential.[21] When two materials of different chemical compositions are joined at an interface, three distinct mechanisms may contribute to a decrease in system energy and thereby promote bond formation: 1) elimination of a high-energy interface, 2) reduction in energy due to bond formation at the interface, and 3) reduction in compositional gradient energy through interdiffusion. A free surface has an excess free energy,[11] usually termed surface energy, which is structurally related to unfilled coordination polyhedra. The act of eliminating two such surfaces by bringing the surface atoms of two pieces of metal into close proximity decreases total energy considerably. This mechanism accounts for the diffusion bonding of two similar metals—titanium for example—or of two insoluble materials.

If unlike soluble metals are joined, then the second and third mechanisms above become important. From solution theory the interaction energy[22] E_i for two species of atoms A and B is given by $E_i = E_{AA} + E_{BB} - 2E_{AB}$, where E_{AA}, E_{BB}, and E_{AB} are the atomic interaction energies. If $E_i > 0$ each species of atom tends to bond to its own kind. Hence there is an energy increase with solution and the resulting solubility is small. Conversely when $E_i < 0$ each type of atom can easily bond to the other type, there is an energy

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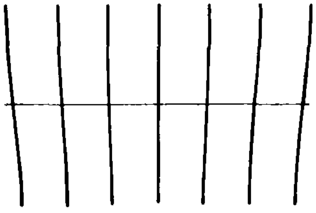
† In some cases metal-oxide or oxide-oxide bonds may result from a high content of insoluble oxides at the surfaces.

decrease with solution, and good solubility is the result. Therefore, A-B bonding at interfaces is enhanced by the same energetics which promote mutual solubility, although the two phenomena (solubility and bonding) are results of a common cause, not cause and effect. Interdiffusion does occur as a means of increasing the number of A-B bonds, or, alternatively, as a means of decreasng the component of free energy associated with the gradient in composition, the third mechanism above. Generally, then, bonding occurs at the interface, and interdiffusion does not contribute to bond properties except by decreasing interfacial stress gradients during subsequent loading of the composite. Once strong bonding at an interface is achieved, failure is often controlled by preexisting flaws in either component or by those produced during the bonding process.[23]

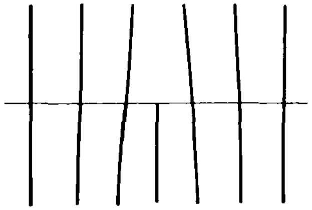
In most cases where favorable bonding energetics are present, strong bonding can be produced in fabricated metal-matrix composites.[24] For such cases, the major difficulty in the bonding cycle is the production of good contact between the components, because of the presence of pores, oxide, or other contaminants. The principal obstacle to many applications is that the interdiffusion reaction cannot be halted once bonding is complete, so further interaction occurs during elevated-temperature utilization of the composite.[25] Since the properties of metal-matrix composites generally become most attractive on a strength-to-weight basis at elevated temperatures, such exposure is inevitable for most composites of this type. Many high strength (or modulus) fibers considered promising for technical applications are chemically unstable in common matrix materials,[26] resulting in fiber solution or the growth of a brittle intermetallic compound harmful to mechanical properties. Eutectic composites circumvent this problem while retaining strong bonding and good high temperature properties, as is discussed below.

TYPES OF INTERFACES

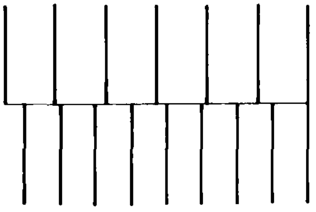
Interfaces between crystalline solids may be categorized[27] as coherent, semicoherent, or noncoherent. Interfaces in eutectic composites produced from the melt may be of any of these types. Liquid-phase-infiltrated or solid-state-bonded composites are usually of the noncoherent-interface type. Models and theories of these types of interfaces were developed well before general interest in composites, as the interfaces are important in monolithic metals containing features such as precipitates[28] or grain boundaries.[29] In Figure 2 the three types of interface are illustrated schematically. All atomic planes are continuous across a coherent interface. If the relaxed spacing of the planes is different, then an elastic compatibility strain



COHERENT



SEMI COHERENT



NON COHERENT

FIGURE 2 Schematic drawing of the three types of interfaces possible between crystalline materials.

exists near the interface. When most planes match with a few extra half planes on one side of the interface, the interface is termed semicoherent. The extra half planes are best thought of as edge dislocations at the interface. An interface at which few if any planes are continuous is called non coherent. Here no elastic coherency strains are present. Detailed dislocation-theory models for interfaces were developed as long ago as 1950 for semicoherent interfaces,[30,31] *and more recently a generalized theory in which all three* types are treated as cases in a continuum of increasing atomic mismatch has been formulated.[32] The energy of interfacial boundaries increases with increasing atomic mismatch, as shown in Figure 3.† An interface of the coherent type is lowest in energy, its energy increasing with elastic mismatch strain. As the mismatch increases past a critical value, the nucleation of dislocations becomes energetically favorable as a means of relieving the elastic compatibility strain. The interfacial dislocation spacing decreases with increasing mismatch, until the interface reaches such a state of disorder that it may be termed noncoherent. These considerations are important to eutectic composites, as discussed below, because their fine reinforcement spacing and growth from the melt allow rapid adjustment of interfaces to achieve low system energy.

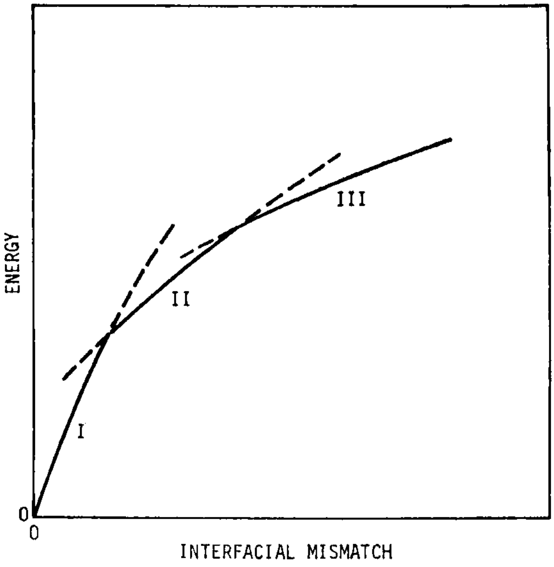


FIGURE 3 Schematic drawing of energy versus mismatch for the three kinds of interfaces.

RELATION OF INTERFACES TO COMPOSITE USAGE

The free energy of a composite system is calculated by adding the volume free energy of the components plus that due to surfaces. Equilibrium is

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† This curve is intended to be schematic; the cusps which may occur at particularly favorable orientations are not shown.

achieved when the sum is minimized. The system is said to be in chemical non-equilibrium if the volume free energy (neglecting electrical, magnetic, etc. fields) is much greater than the equilibrium value, thus providing a strong driving force for chemical change. Similarly, physical nonequilibrium is found when the total surface free energy is much greater than the equilibrium value. In most fabricated metal-matrix composites the importance and magnitude of chemical nonequilibrium exceed those of physical non-equilibrium, while the reverse is true for eutectic composites. Consequently, the greatest potential problem for fabricated composites is degradation due to inter-diffusion, while eutectic composites are susceptible to reinforcement morphology changes to achieve minimum interfacial energy. The degradation kinetics are such that elevated temperature exposure can cause either type of composite to undergo significant changes during service.

The role played by interfaces in metal-matrix composites depends greatly on the fabrication technique employed, as well as the subsequent applications, since the approach toward equilibrium is governed largely by interfacial processes. To amplify upon this statement, some composites and processes will be discussed in detail. For this purpose an idealized phase diagram of a fiber (*N*)—matrix (*M*) system has been drawn in Figure 4. Like many of

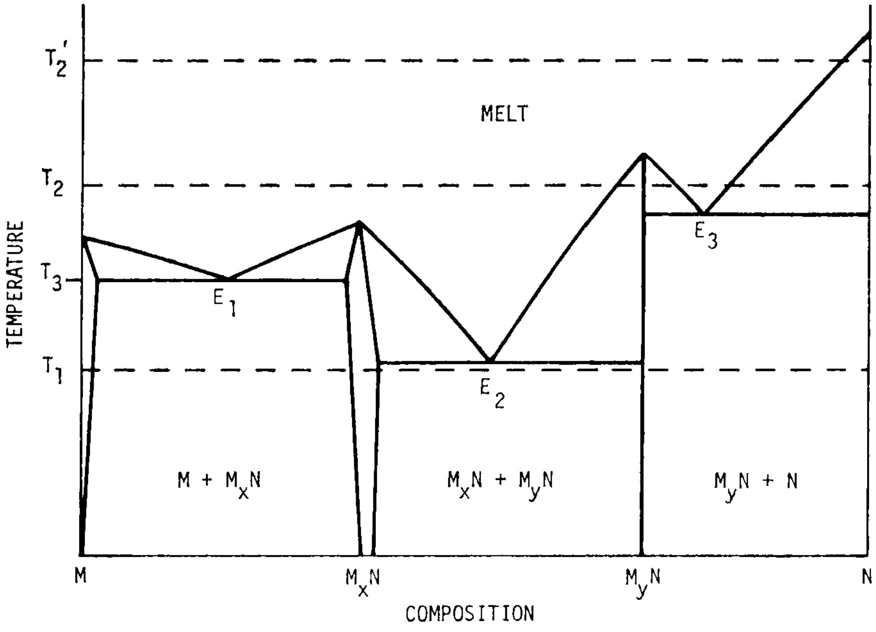


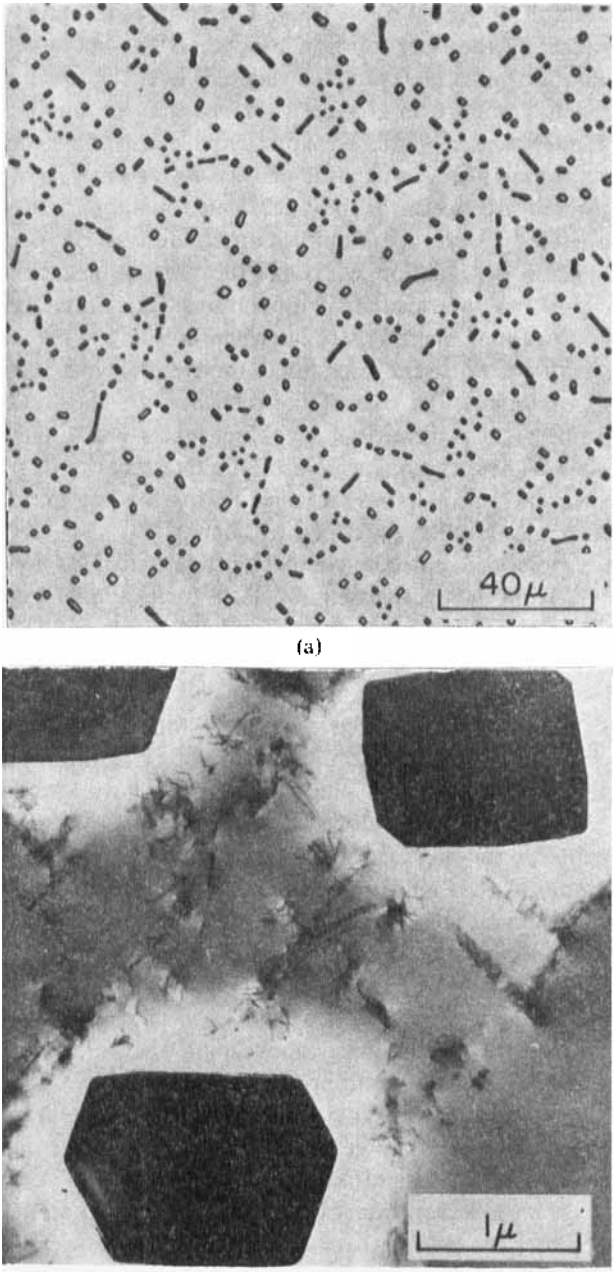
FIGURE 4 Hypothetical phase diagram (temperature vs. composition) for metal-fiber system.

the potentially useful high temperature systems, intermediate phases (semi-ductile M_xN and brittle M_yN) are present, along with three eutectic transformations. Fabricated composites may be formed by bonding N fibers with the M matrix. Eutectic composites may be formed by directional solidification of compositions E_1, E_2, or E_3.

An M-N solid-state diffuson-bonded composite may be formed by pressing or rolling the components at a temperature of T_1 or less. If T_1 is exceeded, a liquid phase can result in fabrication or service. Because T_1 is a low homologous temperature for both M and N, high bonding pressures may be necessary. If the fibers are not broken by the bonding process, and a strong bond can be formed at the interface, the resulting composite may be technically useful. Recent research[24] dealing with transverse tensile tests of such composites shows that, in several aluminum-based-matrix systems, fiber-matrix interface failure does not occur. Failure progresses through the fiber or the matrix, depending on the relative strength of the two. Longitudinal tensile tests[33] often show fiber pullout, indicating that fracture energy is expended by that mechanism. Therefore the interface in those cases seems to be performing its desired function quite well. However, systematic studies of the effect of bond strength in dead loading, fatigue and crack propagation situations have yet to be done. Looking again at Figure 4 and the bonding process at T_1, it is seen that intermetallics M_xN and M_yN may be formed by prolonged exposure. Such exposure at T_1 may occur either in improper bonding or service situations. The brittle M_yN may seriously decrease the fiber fracture strain, thus weakening the composite. The presence of TiB_2 formed in the interdiffusion of Ti and B seriously degrades the B fibers,[25] while the TiC formed between C and Ti is not damaging to fiber strength.[34] The use of interfacial diffusion barriers such as SiC coatings on B fibers[35] usually hinders potential composite degradation, but much research is still being done on the problem.

Matrix-liquid-phase bonding may be done at temperatures such as T_2 or T_2'. At T_2 the brittle intermetallic A_yB can form, but at T_2' it is absent. However, at T_2' the fiber may dissolve rapidly due to its solution kinetics, as in the solution of graphite in nickel. For such liquid phase methods the liquid must wet the fiber so that a bond may be formed, but (especially in the case of ceramic fibers) this wetting may be difficult to achieve due to the already-low surface energies.[36] No pure elements, for example, simultaneously wet, but do not react with, graphite fibers.[12] The possibility of fiber degradation is present, as in the solid-state-bonded case, and the rates are higher at high temperatures. Often the degradation mechanisms are peculiar to the systems under study. For example, high strength tungsten fibers recrystallize in the presence of nickel,[37] greatly decreasing their fracture strain and strength. Some workers[38] have proposed this mechanism for the degradation of graphite in nickel. The composite matrix properties may be degraded by the fiber-solution process. Also, since the possibility of fiber degradation must always be considered, heat treatment procedures ordinarily used to optimize matrix performance may be hindered in these composites. Under certain thermodynamic conditions[39] fibers may oxidize *in situ*, as for graphite fibers in metallic matrices. Thus, in general, chemical instability is of major concern for most fabricated metal-matrix composites, since they are formed in non-equilibrium conditions. It is likely that diffusionally related degradation will limit the use of most composites of this type to low and intermediate temperatures. The interfaces in these types of composites are generally strong (when bonding is reached) and of the non-coherent type. Since the fibers used are relatively large (on the order of 10 microns or larger), the role of the interfaces in plastic deformation is apparently not great. Dislocation blocking effects are minimal because of the large scale. Thus, the major role of the interfaces is to serve as the medium of shear and normal stress transfer during loading and as a site of intermetallic formation and stress concentration.

Directionally solidified eutectic composites[40] are formed by a process entirely different from those discussed above. If, for example, a composition E_1, Figure 4, is solidified in a temperature gradient such that the solid-liquid interface is planar and moves uniformly along the ingot, a phase separation of the form Liquid $\rightarrow M$ (at T_3) $+ M_xN$ (at T_3) occurs. One phase is aligned as rods or plates whose long axis is parallel to the solid-liquid interface's direction of motion. Strong phases may then act as reinforcement in the composite sense. The scale of the phases is very small—0.5 to 5.0 microns typically. A representative structure of such a composite is shown in Figure 5. The interphase interface of this composite is formed from the melt at near equilibrium. As a result surface energy and anisotropy effects become important in determining phase morphology. The interfaces may be coherent, semi coherent, or non coherent, depending upon the system. The interfaces in this type of composite are more closely related to grain boundaries in conventional multiphase metallic alloys than to boundaries in other types of metal-matrix composites discussed above, and they have many of the same virtues and limitations of grain boundaries. The interfaces of eutectics are quite strong—no examples of interface failure and only one of sliding[41] are known to this writer. Exposure to elevated temperatures (near T_3) does not cause growth of any undesired intermetallic, since initially the as-grown composite is in chemical equilibrium at the eutectic melting point. Therefore, eutectic composites have potential for use in the temperature range above that in which most fabricated composites are practical. Due to the small scale of the phases, however, there is a large physical nonequilibrium and driving force for coarsening. Marked reinforcement coarsening is often



(a)

(b)

FIGURE 5 Structure of Ni-W eutectic composite (a) light, and (b) transmitted electron illumination.

observed in these composites,[42] but its effect on properties is not well known. The internal surface area per unit volume for a composite of 25 volume percent of 2 micron diameter fibers is approximately 5×10^3 cm^2 per cm^3, a factor 60 times that of the same volume fraction of 5 mil fibers. Reactions which are dependent on the amount or proximity of surface are more important in eutectics than in composites with larger fibers. For example, precipitation near an interphase boundary in an Al-Cu-Mg eutectic composite[43] is shown in Figure 6. Near the boundary no precipitates are seen, as the vacancies needed in the process of precipitation diffuse to the boundary, leaving a deficient zone. Here, then, the small composite scale coupled with the nature of the interfaces affects matrix properties directly. The interphase boundaries also control composite properties indirectly by determining morphology, phase transformations, deformation, etc. Major barriers to the use of eutectic composites are a general lack of understanding of their properties, newness of the field, inability to form some shapes, often-low ductility, and a scarcity of high-temperature applications. They are, however,

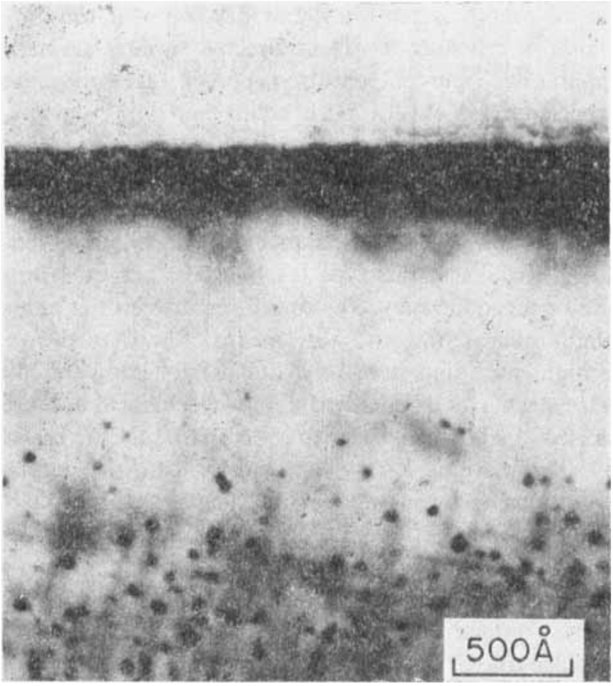


FIGURE 6 Precipitation in Al-rich phase near interphase boundary of Al-Cu-Mg eutectic composite (ref. 43).

under serious consideration for gas turbine,[44] rocket engine, and non-mechanical applications.

EXPERIMENTAL TECHNIQUES

For fabricated metal-matrix composites formed with the matrix in the liquid phase, wettability studies combined with reaction product and rate studies[45] have proved useful as screening and development techniques. A good deal of research in this area has been directed toward the mechanics of fabrication apparatus. Time-temperature-property fabrication and exposure tests have been carried out for both solid- and liquid-matrix-phase fabricated composites,[46] with property evaluation primarily by quasi-static, fatigue, or creep loading. Matrix hardness tests have been used to study matrix strength as a function of distance from the interface,[47] but no analytical studies have established the changes in stress state under the indenter near a fiber. Results of this type test must be accepted only with reservation because of this deficiency. The interface between the phases was first studied by light microscopy and this means is still used for some applications. The high resolution necessary and the inherent difficulties in polishing multiphase structures of greatly different mechanical and chemical properties has caused a shift in the major emphasis to methods using electron or X-ray illumination. X-ray techniques[48] can give information on chemical compositions or strains present, but features cannot be located or sized accurately. Replica electron microscopy is used to provide some information on the geometry of intermetallics, but it cannot be used for chemical identification. Replica studies of fracture surfaces give considerable information when the surfaces are fairly flat, with no fiber segments projecting from the surface to tear the replica as it is removed.[49]

These techniques have been largely supplanted by more sophisticated methods in recent years. Chemical thinning techniques allowed the production of thin foils of eutectic[50] and all-metal fabricated[51] composites about five years ago, and now transmission electron microscopy is routinely used to study their microstructures. Recently ion thinning[52] has broadened the scope of the method to include fabricated composites with boron and carbon fibers. Morphology, crystallography, and to some extent chemistry[49] can be determined through the use of imaging and selected-area-diffraction techniques. Scanning electron microscopy[53] is now generally used both for interface and fracture surface research, although the interface applications are still largely in the qualitative stage. The electron microprobe,[54] Auger electron spectroscopy,[55] and low-energy electron diffraction[56] are used for various chemical compositon and orientation studies of composites and interfaces. These techniques are being constantly refined into more useful methods available to the materials researcher.

Summary

1. Strong interfaces may be produced by liquid- or solid-state bonding in fabricated metal-matrix composites. These interfaces generally are not directly the sources of failure, although failure may originate in the near-interface region. Before manipulation of interfacial properties can become a reality, methods for controlling interphase reaction problems must be developed.
2. Directionally solidified eutectic composites offer great potential for high temperature applications. The interfaces are strong, and the fine scale of reinforcement and large amount of interfacial area can influence morphology, stability, deformation, etc., greatly.
3. Since metal-matrix composites are used at elevated temperatures, the temperature and environment dependence of properties near the interface is important. Response to gaseous environments remains a key problem because of possible degradation of the components.

Acknowledgement

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