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High Temperature Structural Silicides

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Abstract:

Structural silicides have important high temperature applications in oxidizing and aggressive environments. Most prominent are $MoSi_2$-based materials, which are borderline ceramic-intermetallic compounds. $MoSi_2$ single crystals exhibit macroscopic compressive ductility at temperatures below room temperature in some orientations. Polycrystalline $MoSi_2$ possesses elevated temperature creep behavior which is highly sensitive to grain size. $MoSi_2$-Si_3N_4 composites show an important combination of oxidation resistance, creep resistance, and low temperature fracture toughness. Current potential applications of $MoSi_2$-based materials include furnace heating elements, molten metal lances, industrial gas burners, aerospace turbine engine components, diesel engine glow plugs, and materials for glass processing.

Introduction:

Over the past few years, structural silicides have emerged as important elevated temperature structural materials for applications in oxidizing and aggressive environments (1-3). There are a relatively large number of silicides know, most of which have seen little characterization of their properties (4). For structural purposes, the silicides of most interest are the transition metal silicides, and more particularly molybdenum-based silicides.

In terms of structural materials that can be used in oxidizing environments, there is a temperature cutoff point at approximately 1000 °C. Below this temperature materials such as nickel and cobalt-based superalloys can be employed, as well as materials such as titanium and nickel aluminides and SiC fiber reinforced glass-ceramics. However, above 1000 °C, for oxidation-resistance and elevated temperature strength reasons, one must resort to materials such as the silicon-based structural ceramics Si_3N_4 and SiC, advanced high temperature intermetallics such as NiAl and $NbCr_2$, and the class of structural silicide materials, most notably $MoSi_2$.

Potentially interesting and important structural silicides are shown in Table I. $MoSi_2$ has been by far the most investigated due to its excellent oxidation resistance. WSi_2 forms a complete solid solution with $MoSi_2$. Mo_5Si_3 is adjacent to $MoSi_2$ in the Mo-Si phase diagram. $NbSi_2$ and $TaSi_2$ have exhibited single crystal ductility in some orientations at temperatures near room temperature. Ti_5Si_3 has a high melting point and low density.

Table I: Some Interesting Potential Structural Silicides

Silicide	Melting Point (°C)	Crystal Structure	Density (g/cm^3)
$MoSi_2$	2030	Tetragonal	6.24
WSi_2	2160	Tetragonal	9.86
$NbSi_2$	1930	Hexagonal	5.66
$TaSi_2$	2200	Hexagonal	9.10
$TiSi_2$	1500	Orthorohombic	4.04
$CrSi_2$	1490	Hexagonal	4.98
$CoSi_2$	1326	Cubic	4.95
YSi_2	1835	Rhombohedral	4.50
Mo_5Si_3	2160	Tetragonal	8.24
Ti_5Si_3	2130	Hexagonal	4.32

Characteristics of $MoSi_2$:

As has been indicated, $MoSi_2$ has received the most attention to date in the field of structural silicides. It is pertinent to ask the question: What is $MoSi_2$? In order to answer this question, it is first necessary to address the question: What is a ceramic? Although there have been many definitions of a ceramic put forward, a definition which is both comprehensive and succinct is the following (1): A ceramic is a solid, iono-covalent, inorganic compound. Based on this definition of a ceramic, $MoSi_2$ is then a borderline ceramic-intermetallic

compound, since its atomic bonding is a combination of both covalent and metallic (5).

It is the collection of properties of $MoSi_2$ that make it interesting as a high temperature structural material (1). $MoSi_2$ has a high melting point of 2030 °C. It has superb high temperature oxidation resistance, essentially equivalent to that of SiC since it forms a thin coherent and adherent protective silica layer. In polycrystalline form, $MoSi_2$ exhibits a brittle-to-ductile transition in compression in the vicinity of 1000 °C or lower, although some orientations of single crystals actually show macroscopic ductility at much lower temperatures. The material is thermodynamically stable with a wide range of structural ceramics, including Si_3N_4, SiC, Al_2O_3, ZrO_2, mullite, TiB_2, and TiC. Thus, there is a significant potential for composite development. It can also be alloyed with other high melting point silicides such as WSi_2, $NbSi_2$, Mo_5Si_3, and Ti_5Si_3. Due to the metallic nature of its bonding, $MoSi_2$ can be electro-discharge machined, thus making it easier to machine than most structural ceramics. Finally, $MoSi_2$ is an abundant, relatively low cost material, which is also environmentally benign.

In terms of engineering properties, $MoSi_2$ has a thermal expansion coefficient close to that of Al_2O_3. Its thermal conductivity is intermediate between that of Si_3N_4 and SiC. The elastic modulus of $MoSi_2$ is close to the elastic modulus of SiC. While the high temperature oxidation resistance of $MoSi_2$ is similar to that of SiC, maximum oxidation rates in $MoSi_2$ actually occur at the intermediate temperature of 500 °C.

Single Crystal $MoSi_2$:

The crystal structure of $MoSi_2$ is tetragonal. The compressive deformation of single crystal $MoSi_2$ has been investigated as a function of temperature (6-8). Figure 1 shows the compressive yield stress of $MoSi_2$ single crystals as a function of temperature. There are two key points to be made about the data in Figure 1. First, elevated temperature strength in the [001] orientation is very high, essentially similar to that of c-axis sapphire. This suggests the potential for crystallographic texture development to increase high temperature mechanical strength, such as polycrystalline fiber or wire development with a [001] texture. The second key observation is that macroscopic compressive ductility occurs in some crystallographic orientations at temperatures as low as -100 °C. This presents the possibility of significantly lowering the ductile-to-brittle transition temperature of $MoSi_2$ by suitable alloying.
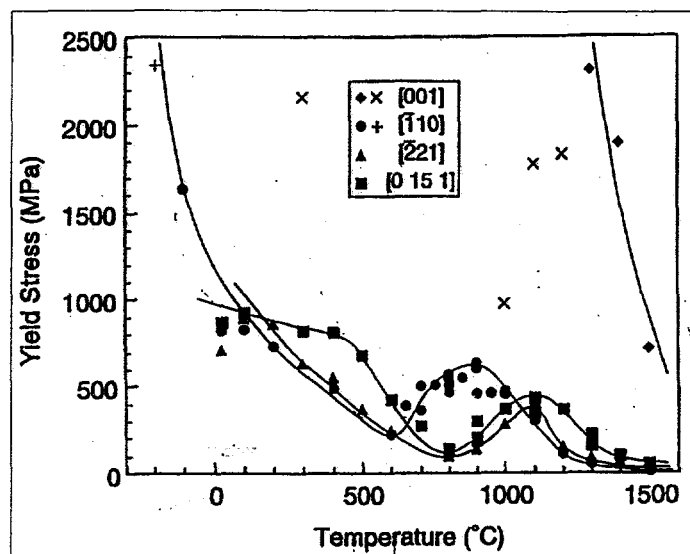
Figure 1: Compressive yield stress of MoSi₂ single crystals as a function of temperature (8).

The observed slip systems in MoSi₂ are {013}<331>, {110}<111>, {011}<100>, {010}<100>, and {023}<100> (6-8). An anomalous behavior is observed for the {013}<331> slip system, in that this slip system operates only at high temperatures for crystallographic orientations near [001], yet is observed to be operative at low temperatures for other crystallographic orientations (8). This unusual behavior is likely due to dislocation core effects. If the {013}<331> slip system can be made to operate in the [001] orientation, then there is a significant possibility of markedly lowering the brittle-to-ductile transition of polycrystalline MoSi₂ (8). The alloying of MoSi₂ to promote this slip system is presently an active area of silicide research.

Figure 2 shows a room temperature Vickers indentation in single crystal MoSi₂ oriented in the [001] direction (9). Both indentation cracks and dislocation slip lines are observed on the polished surface at room temperature. This indicates that MoSi₂ is a semi-brittle material somewhat analogous to MgO. The direction of the indentation cracks suggests that {100} planes may be cleavage-type planes in MoSi₂. If the slip trace planes are perpendicular to the plane of polish, then slip on {100} and {110} planes at room temperature is suggested.
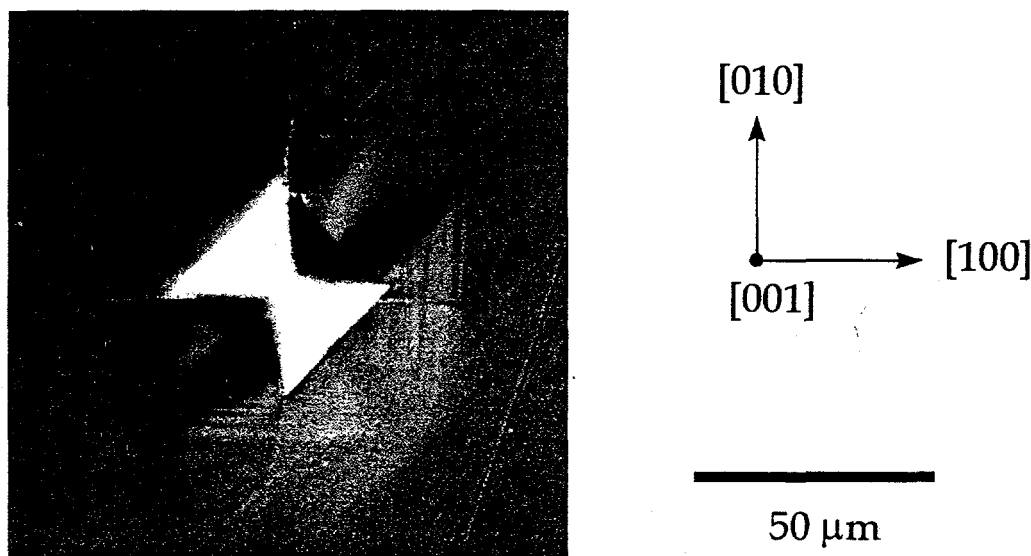
Figure 2: Room temperature 1000 gm Vickers indentation in single crystal MoSi₂ (9).

Polycrystalline MoSi₂:

At the present time, polycrystalline $MoSi_2$ is brittle at room temperature, with a polycrystalline fracture toughness of approximately 3 MPa $m^{1/2}$ (10). It has been shown that at room temperature, the fracture mode of $MoSi_2$ is 75% transgranular and 25% intergranular (10). The brittle-to-ductile transition of large grained, low oxygen content $MoSi_2$ has been investigated in both compression and bending (11). While a BDTT of approximately 1000 °C is seen in compression, the BDTT in bending occurs at approximately 1350 °C in bending. The BDTT of polycrystalline $MoSi_2$ is sensitive to both grain size and oxygen content, decreasing with decreasing grain size and increasing oxygen content. It has been shown that prestraining at 1300 °C can lower the BDTT of polycrystalline $MoSi_2$ to appromately 750 °C (12). This is due to the introduction of mobile dislocations by the high temperature prestraining treatment.

The elevated temperature creep resistance of polycrystalline $MoSi_2$ is highly sensitive to grain size (13). This effect is shown in Figure 3. The creep resistance of $MoSi_2$ has been shown to increase by three orders of magnitude with only an 11 µm increase in the grain size. A full explanation of this grain size sensitivity has not yet been obtained. However, the elevated temperature creep mechanisms in polycrystalline $MoSi_2$ are known to be a combination of grain boundary sliding and dislocation plasticity (13). This grain size effect on creep

resistance is important from the viewpoint of composite design for optimized creep properties.
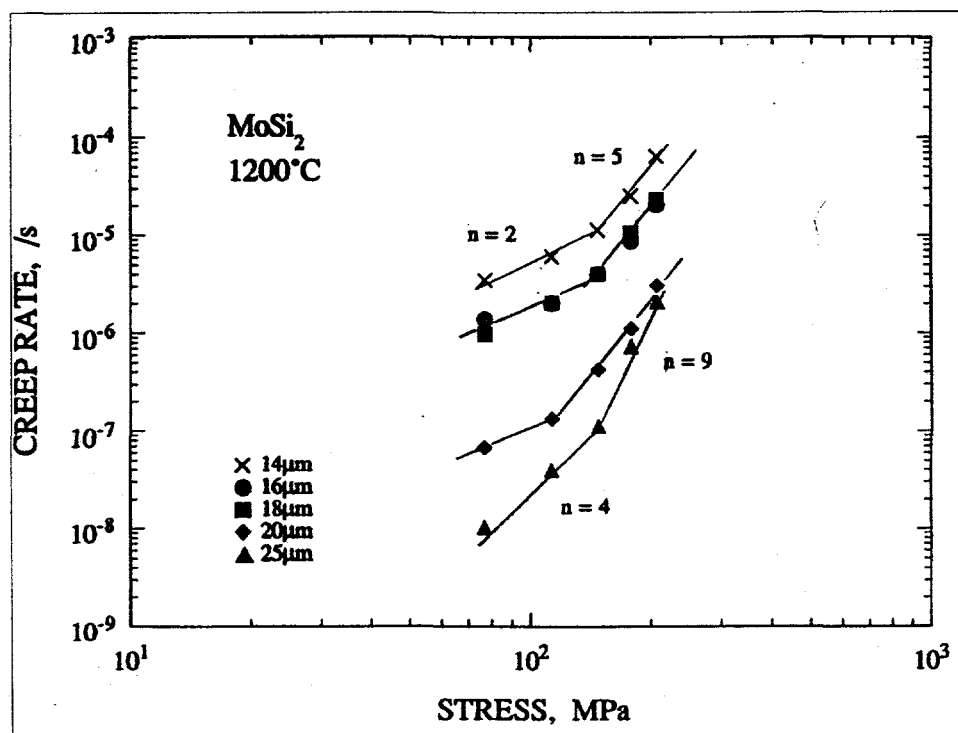

Figure 3: Effect of grain size on the creep of polycrystalline MoSi₂ (13).

MoSi₂-based Composites:

For MoSi₂-based materials to be successfully employed in high temperature structural applications, both the high temperature creep resistance and the low temperature fracture toughness must be improved. The composite approach with ceramic reinforcements has been shown to yield such improvements, with no significant reduction in the oxidation resistance of these materials (1). Composites may be a MoSi₂ matrix reinforced with ceramic, or a ceramic matrix reinforced with MoSi₂.

Both SiC whisker and SiC particle reinforcements have been observed to significantly increase the elevated temperature creep resistance of MoSi₂-based materials, and moderately increase the room temperature fracture toughness (14-16). The addition of ZrO₂ particles to a MoSi₂ matrix can produce significant transformation toughening effects in MoSi₂ composites, with the maximum toughening observed for unstabilized ZrO₂ particles (17).

MoSi$_2$-Si$_3$N$_4$ Composites:

The MoSi$_2$-Si$_3$N$_4$ composite system is a very interesting and important one. MoSi$_2$ and Si$_3$N$_4$ are thermodynamically stable compounds (18). Additions of Si$_3$N$_4$ to a MoSi$_2$ matrix significantly improve the intermediate temperature oxidation resistance of MoSi$_2$ and its elevated temperature mechanical properties (19). Additions of MoSi$_2$ to a Si$_3$N$_4$ matrix allow for the electro-discharge machining of Si$_3$N$_4$, and also lead to improved fracture toughness and elevated temperature oxidation characteristics (20,21,22). Figure 4 shows the effects on room temperature fracture toughness and elevated temperature creep resistance of additions of MoSi$_2$ to a Si$_3$N$_4$ matrix (21).
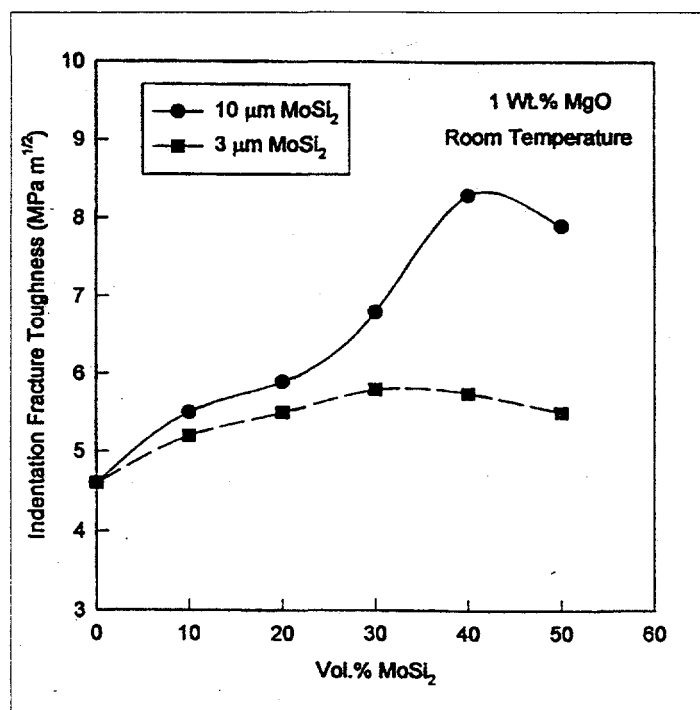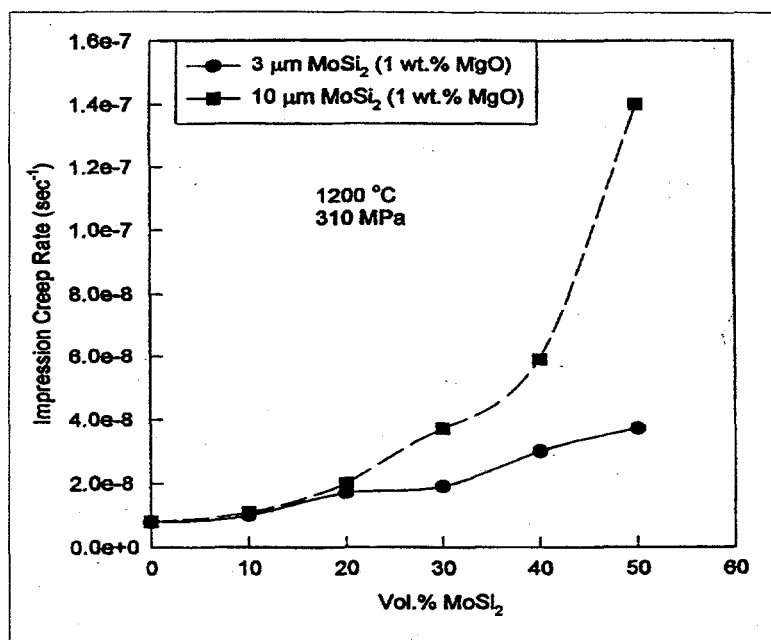
Figure 4: Room temperature fracture toughness and elevated temperature creep resistance of MoSi₂ particle-Si₃N₄ matrix composites (21).

Boron-Mo₅Si₃:

The silicide compound Mo_5Si_3 is adjacent to $MoSi_2$ in the Mo-Si phase diagram, and has a high melting point of 2160 °C. A major drawback to its application has been the fact that its oxidation resistance is greatly inferior to that of $MoSi_2$ (2). Recently, it has been shown that small additions of boron to Mo_5Si_3 significantly improve the oxidation resistance of this material (23). These small additions of boron lead to the formation of a protective borosilicate glass oxidation layer. Additionally, the boron additions produce the formation of a multiphase composite material consisting of $Mo_5Si_3B_x$, $Mo_5(SiB)_3$, Mo_3Si, $MoSi_2$, and MoB, with the exact phase composition depending on the level of boron addition. The boron-Mo_5Si_3 materials have been reported to possess good elevated temperature creep resistance (24).

Applications of MoSi₂-based Materials:

MoSi₂-based materials are beginning to develop a broad base of important industrial applications. These applications are driven by the elevated temperature

mechanical properties of these materials, in combination with other properties such as electrical conductivity and oxidation/corrosion resistance.

Heating Elements:

$MoSi_2$ materials have been employed for a number of years as heating elements for air furnaces. The recent Kanthal Super 1900 heating elements can operate at an element temperature of 1900 °C in air and oxidizing environments. These Super 1900 elements are actually a solid solution alloy of $MoSi_2$ and WSi_2. The major problems with the $MoSi_2$-based heating elements are brittle fracture, which makes the elements difficult to handle, and high temperature creep, which causes the elements to deform. Both of these aspects limit element life, as well as affect furnace design. Current elements are U-shaped and typically hung vertically due to limitations in mechanical properties. As both the fracture toughness and creep resistance of $MoSi_2$-based materials continue to improve by composite and alloying approaches, there will be significant effects on enhanced furnace element lifetime as well as greater flexibility in furnace design.

Molten Metal Lances:

A number of foundry operations require that gases be injected into molten metals. Microlaminate $MoSi_2$-Al_2O_3 composite tubes were fabricated by plasma spray forming (25). These tubes were then tested as inert gas lances in molten aluminum alloy at 725 °C and molten copper at 1200 °C. The $MoSi_2$-Al_2O_3 composites performed very well in both molten metals (25). In the case of the molten copper, alternative tube materials of graphite and SiC were also tested for comparison. The graphite tube lasted for only fifteen minutes in the molten copper, while the SiC tube thermal shocked upon immersion in the molten copper. In contrast, the $MoSi_2$-Al_2O_3 microlaminate tube withstood four hours in the molten copper and could have lasted longer, however the test was terminated at the four hour mark. The composite tube was resistant to chemical attack by the molten copper due to the presence of the Al_2O_3 phase, while it exhibited thermal shock resistance and "graceful failure" type mechanical behavior due to plastic deformation of the $MoSi_2$ phase (25).

Industrial Gas Burners:

The industrial gas burner industry is in the process of developing gas burners which will burn oxygen-natural gas mixtures rather than air-natural gas mixtures, in order to reduce NO_x environmental emissions. Because such

oxygen-natural gas burners must operate at higher temperatures than air-natural gas burners, there is a need to develop new burner materials which are resistant to the oxygen-natural gas combustion environment. Studies have shown that $MoSi_2$ possesses significant resistance to oxygen-natural gas combustion at high temperatures (26). After an initial transient period, material stability is achieved through the formation of a stable Mo_5Si_3 layer. Stability occurs under both stoichiometric and fuel-rich combustion conditions. Prototype $MoSi_2$ gas burners have been fabricated by a plasma spray forming process (27).

Aerospace Gas Turbine Engines:

Pratt & Whitney has been developing advanced materials for a blade outer air seal (BOAS) hot section component of its gas turbine engines. In the engine, the BOAS is a stationary part which is located directly opposite of the rotating hot section turbine blades. The purpose of the BOAS is to maintain a small gap of stable dimensions between itself and the turbine blade. If this gap widens during operation of the turbine, it directly affects the turbine efficiency. Although stationary, the BOAS is exposed to high turbine gas temperatures and significant thermal stresses.

Gas burner testing by Pratt & Whitney has shown that $MoSi_2$-SiC and $MoSi_2$-Si_3N_4 composites possess significant thermal shock resistance in the simulated jet fuel combustion environment (28). These materials survived 250 cycles from room temperature to 1500 °C with no failures. Recent work at NASA-Lewis has concentrated on $MoSi_2$-Si_3N_4 composites reinforced with SiC continuous fibers. Thermomechanical Charpy impact tests as a function of temperature (which are designed to evaluate the material resistance to foreign impact damage in the engine) have demonstrated that these composite materials absorb significant impact energy at both room temperature and elevated temperatures (29).

Diesel Engines:

$MoSi_2$-Si_3N_4 composite diesel engine glow plugs have recently been developed by Toyota Central R&D in Japan (30). The $MoSi_2$-Si_3N_4 glow plugs contain 30-40 vol.% $MoSi_2$ phase in a Si_3N_4 matrix. These glow plugs have two distinct practical advantages over metal glow plugs. First, they are highly resistant to the diesel fuel combustion environment and thus have a long lifetime of approximately thirteen years. Second, they can be heated at higher heating rates with the result that the diesel engine can be started faster. An inner composite cylinder with an interconnected $MoSi_2$ phase provides the necessary

electrical conductivity for the glow plug application. An outer, non-conducting sheath of the same $MoSi_2$-Si_3N_4 phase composition is employed as a cover for the inner conducting composite and has the same thermal expansion coefficient and thermal conductivity. This microstructural tailoring of the $MoSi_2$-Si_3N_4 composite allows for optimum performance of the diesel glow plug.

Glass Processing:

At the present time, metals and ceramic refractories are primarily employed in applications and components requiring contact with molten glasses. The refractory metal molybdenum is highly resistant to corrosion when immersed in molten glass. However, due to its poor oxidation resistance, it cannot be employed at or above the molten glass line. The precious metal platinum is also employed in contact with molten glasses, but this material is very expensive. The AZS multiphase refractory ceramic (Alumina-Zirconia-Silica) is also used for containing molten glasses, but suffers from relatively poor mechanical properties.

Recently, it has been shown that $MoSi_2$ is also a material which is quite resistant to corrosion by molten glasses (31-33). $MoSi_2$ shows excellent corrosion resistance below the glass line due to Mo_5Si_3 formation, and excellent oxidation resistance above the glass line due to SiO_2 formation. While corrosion rates of $MoSi_2$ are somewhat higher at the glass line than below the glass line, it has been reported that anodic protection of the $MoSi_2$ significantly lowers corrosion rates at the glass line (33).

This corrosion resistance of $MoSi_2$ to molten glasses in combination with its elevated temperature mechanical properties has recently lead to the Kanthal Corporation marketing a new $MoSi_2$ immersion tube for the injection of gases into molten glass (34). It has also initiated a Cooperative Research and Development Agreement (CRADA) between the Los Alamos National Laboratory and Schuller International Inc. (35). Schuller International is a major U.S. producer of fiberglass, and the objective of the CRADA is the development of $MoSi_2$-based materials for fiberglass processing applications and components.

other people at Los Alamos who have contributed to the structural silicide research activities there.

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