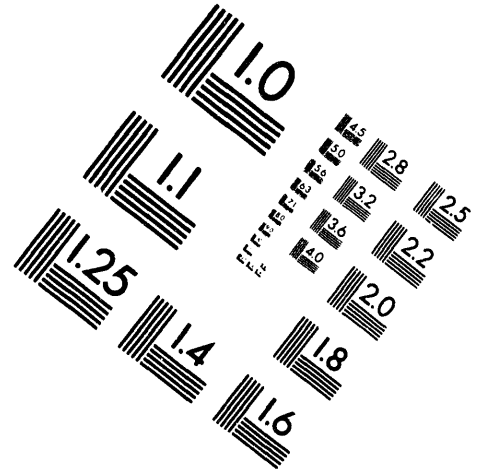
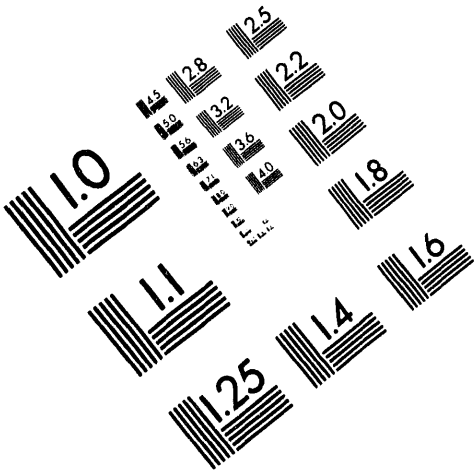




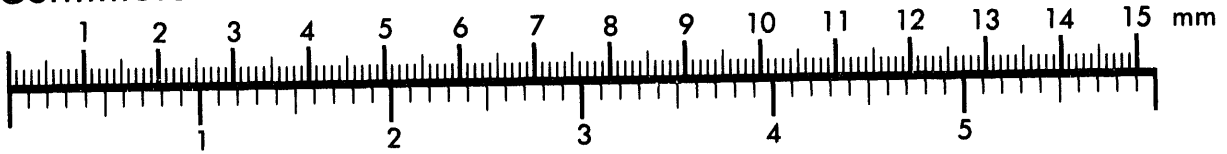
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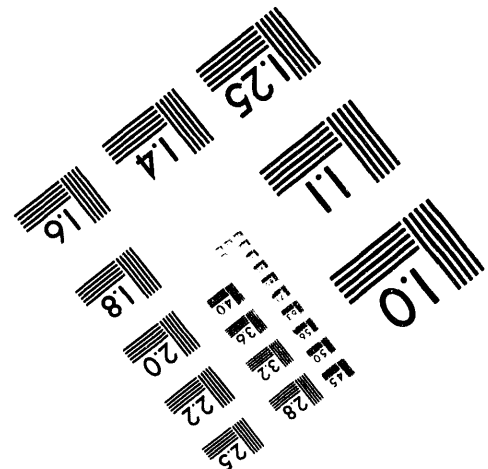
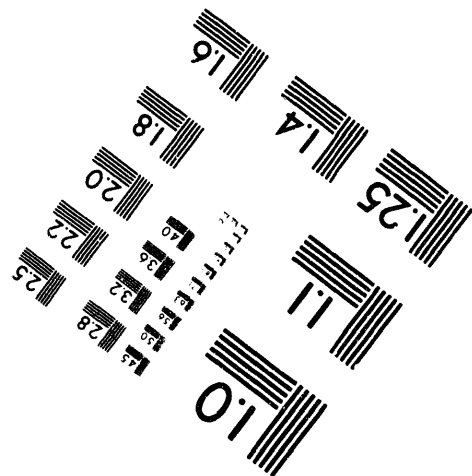
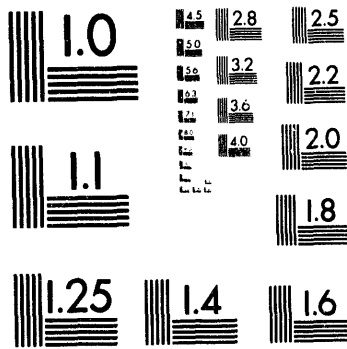
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TITLE: Mechanical Behavior of MoSi<sub>2</sub> and MoSi<sub>2</sub> Composites

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# **MECHANICAL BEHAVIOR OF MoSi<sub>2</sub> AND MoSi<sub>2</sub> COMPOSITES**

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## **ABSTRACT:**

MoSi<sub>2</sub> is a key member of the new class of high temperature structural silicide materials. Important features of the mechanical behavior of MoSi<sub>2</sub> and MoSi<sub>2</sub> composites are reviewed. The mechanical properties of MoSi<sub>2</sub> single crystals, polycrystalline MoSi<sub>2</sub>, and MoSi<sub>2</sub>-based composites are discussed in association with properties such as elevated temperature deformation and low temperature fracture toughness. Interrelationships between single crystal, polycrystal, and composite mechanical behavior are identified.

## **INTRODUCTION:**

Materials based on the silicide MoSi<sub>2</sub> hold considerable promise for a wide variety of elevated temperature structural applications at temperatures above 1000 °C [1-3]. Industrial applications include furnace elements and components, power generation components, high-temperature heat exchangers, gas burners, lances for liquid metals and glasses, igniters, and high-temperature filters. Aerospace applications include turbine aircraft engine hot-section components such as blades, vanes, combustors, nozzles, and seals. Automotive applications involve components such as turbocharger rotors, valves, glow plugs, and advanced turbine engine parts.

MoSi<sub>2</sub> possesses a combination of properties which make it attractive as a high temperature material. It has a high melting point of 2030 °C, with excellent high temperature oxidation resistance, essentially equivalent to the oxidation resistance of the silicon-based structural ceramics SiC and Si<sub>3</sub>N<sub>4</sub>. It exhibits a brittle-to-ductile transition in the vicinity of 900 °C, as determined by the compression of single crystals. MoSi<sub>2</sub> is thermodynamically stable with a wide variety of potential ceramic reinforcements for composites, including ceramics such as SiC, Si<sub>3</sub>N<sub>4</sub>, ZrO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, mullite, YAG, Y<sub>2</sub>O<sub>3</sub>, TiB<sub>2</sub>, and TiC. Its stability extends essentially to the full range of important structural ceramic materials. MoSi<sub>2</sub> can be alloyed with other high melting point silicides as another potential avenue to improve its properties. Possible silicide alloying species are WSi<sub>2</sub>, NbSi<sub>2</sub>, CoSi<sub>2</sub>, Mo<sub>5</sub>Si<sub>3</sub>, and Ti<sub>5</sub>Si<sub>3</sub>. MoSi<sub>2</sub> is an abundant, relatively low cost material which is non-toxic and environmentally benign. Because of its relatively high electrical conductivity, MoSi<sub>2</sub> and MoSi<sub>2</sub>-based materials can be electro-discharge machined (EDM).

There are four general aspects to the structural use of MoSi<sub>2</sub> materials. The first is as a matrix for MoSi<sub>2</sub>-based composites. This aspect has seen the most activity to date. However, a second aspect is the use of MoSi<sub>2</sub> as a reinforcement for structural ceramic matrix composites. Very little work has been done to date in this area. Third, MoSi<sub>2</sub> is a potential high temperature joining material for structural ceramics, due to its oxidation resistance and high melting point. Finally, MoSi<sub>2</sub>-based materials can serve as oxidation-resistant coatings for refractory metals and carbon-based materials.

The silicide MoSi<sub>2</sub> can best be described as a borderline ceramic-intermetallic compound. A generic definition of a ceramic is that a ceramic is a solid, ionic-covalent, inorganic compound. Applying this definition to MoSi<sub>2</sub> indicates its borderline nature, since it differs from a ceramic only because its atomic bonding is a mixture of covalent and metallic. MoSi<sub>2</sub> possesses ceramic-like high temperature oxidation resistance, and metal-like electrical conductivity. This material exhibits ceramic-like brittleness at room temperature, and hence fracture toughness is a major issue. It also shows metal-like plasticity at elevated temperatures, with the result that creep resistance is a major issue.

For MoSi<sub>2</sub>-based materials to be successful in elevated temperature structural applications, it is necessary to improve both high temperature strength and creep resistance, as well as low temperature fracture toughness. In this paper, important aspects of the mechanical behavior of MoSi<sub>2</sub> and MoSi<sub>2</sub> composites are reviewed. This is best accomplished by considering the behavior of single crystals, polycrystals, and composites based on this material.

### **SINGLE CRYSTAL MoSi<sub>2</sub>:**

The crystal structure of MoSi<sub>2</sub> is the C11<sub>b</sub> type, as shown in Figure 1. It can be viewed as the stacking of three body centered sub-unit cells. Unal et.al. [4] and Maloy et.al. [5] have analyzed the possible slip systems in MoSi<sub>2</sub>. Potential slip systems are shown in Table 1. Important burgers vectors for slip are shown in Figure 2. In general, the Peierls stress for a given slip system is expected to decrease with increasing ratio of slip plane spacing/Burgers vector,  $d/b$ . Thus, in Table 1, the slip systems with the highest values of  $d/b$  might be expected to be favored in the deformation of MoSi<sub>2</sub>. However, as will be shown, this is not necessarily always the case for this material.

The mechanical behavior of MoSi<sub>2</sub> single crystals has been studied to a limited extent [5,6-10]. Umakoshi et.al. [7] were the first to determine the compressive deformation of MoSi<sub>2</sub> single crystals as a function of temperature and crystallographic orientation. They determined that the onset of plastic deformation in single crystals began at a temperature of approximately 900 °C. They also showed that crystallographic orientation had a significant effect on yield stress. Specifically, the yield stress of crystals in the [001] orientation was observed to be very much higher than the yield stress in other crystallographic orientations.

Yield stresses of MoSi<sub>2</sub> single crystals as determined by Maloy [5,10] are shown in Figure 3. As with previous work, the onset of plastic deformation appears to occur at 900 °C in MoSi<sub>2</sub> single crystals. Yield strengths in the [001] orientation were observed to be significantly higher than those reported by Umakoshi et.al. [7], with a yield stress value of 600 MPa at 1600 °C observed. Yield stress values in the [001] direction were noted to be

sensitive to the applied strain rate, and increased significantly with increasing strain rate. Strengths in other crystallographic orientations were much lower than those in the [001] direction.

Slip systems which have been observed to operate in elevated temperature deformation of MoSi<sub>2</sub> single crystals are shown in Table 2 [5,10]. At the lower temperatures, the {013}1/2<331> slip system is observed to be active, as well as the slip systems {011}<100> and {110}1/2<111>. At higher temperatures, the {110}, {011}, and {001} slip planes have been observed, with <100>, <110>, and 1/2<111> Burgers vectors.

Nakamura et.al. [9] have determined the elastic stiffnesses and compliance constants for MoSi<sub>2</sub> single crystals. These values are shown in Table 3. From these values, they have estimated the following values for polycrystalline MoSi<sub>2</sub>: Bulk modulus  $K = 210$  GPa, Young's modulus  $E = 440$  GPa, Shear modulus  $G = 191$  GPa, Poisson's ratio  $\nu = 0.151$ .

### **POLYCRYSTALLINE MoSi<sub>2</sub>:**

The strength of polycrystalline MoSi<sub>2</sub> generally depends on the silica content and the grain size of the material. High silica contents and small grain sizes tend to promote grain boundary sliding as a deformation mechanism, which can operate at lower stress levels than dislocation plasticity. The yield stress behavior of low silica content, relatively large grained MoSi<sub>2</sub> polycrystalline material has been studied by Aikin [11], and is shown in Figure 4. Under compressive deformation, polycrystalline MoSi<sub>2</sub> can exhibit significant plastic deformation at temperatures as low as 900 °C. However, in bending deformation, the brittle-to-ductile transition temperature has been observed to be between 1300-1400 °C. As shown in Figure 4, the yield stress of polycrystalline MoSi<sub>2</sub> is in the vicinity of 200 MPa at 1400 °C for both compression and bending modes of mechanical loading.

Gibala et.al. [12] have shown that prestraining polycrystalline MoSi<sub>2</sub> at an elevated temperature of 1300 °C can significantly lower the brittle-to-ductile transition temperature of the material as measured in compression. The prestraining has been observed to lower

the transition temperature to as low as 750 °C. This transition temperature lowering is attributed to the introduction of mobile dislocations into the material as a result of the elevated temperature prestrain. The presence of greater numbers of mobile dislocations then promotes increased plastic deformation at the lower temperatures.

Sadananda et.al. [13] have shown that the elevated temperature creep behavior of polycrystalline MoSi<sub>2</sub> is sensitive to grain size, as shown in Figure 5. This sensitivity of creep rate to grain size in MoSi<sub>2</sub> is higher than expected, since elevated temperature creep appears to be controlled primarily by dislocation glide/climb processes, as well as grain boundary sliding accommodated by dislocation plasticity. The grain size exponent observed for MoSi<sub>2</sub> is in the range of 5-8. For Nabarro-Herring creep one expects a grain size exponent of 2, while for Coble creep a grain size exponent of 3 is expected. These grain size effects on the elevated temperature deformation of polycrystalline MoSi<sub>2</sub> are unusual and not fully understood at the present time. However, they also occur in other intermetallic systems as well [13].

At low temperatures, polycrystalline MoSi<sub>2</sub> is a brittle material that is similar to ceramics in this characteristic. Wade and Petrovic have determined that at room temperature, the fracture mode of MoSi<sub>2</sub> is 75% transgranular and 25% intergranular [14]. The room temperature fracture toughness of MoSi<sub>2</sub> is 3 Mpa m<sup>1/2</sup> [14,15]. This fracture toughness value is similar to that of a polycrystalline, equiaxed Si<sub>3</sub>N<sub>4</sub> ceramic material which is densified without the use of densification aids.

The presence of a silica phase in polycrystalline MoSi<sub>2</sub> has been observed to have a detrimental effect on the fracture toughness of this material, particularly at elevated temperatures [16]. Figure 6 shows fracture toughness as a function of temperature, for polycrystalline MoSi<sub>2</sub> both without and with carbon additions. Without a carbon addition, silica in the MoSi<sub>2</sub> microstructure causes the toughness to decrease with increasing temperature, due to grain boundary fracture effects. However, with carbon additions, the toughness increases, due to the operation of dislocation plasticity. A fracture toughness of nearly 12 Mpa m<sup>1/2</sup> is observed at 1400 °C for polycrystalline MoSi<sub>2</sub> with added carbon to remove the silica phase. A transition from intergranular to transgranular fracture in

polycrystalline MoSi<sub>2</sub> at elevated temperatures has been observed as a result of these carbon additions [16].

### **MoSi<sub>2</sub>-BASED COMPOSITES:**

#### **Creep Behavior:**

The creep behavior of MoSi<sub>2</sub>-based materials has recently been reviewed by Sadananda and Feng [17]. A number of factors have been observed to improve the elevated temperature creep resistance of these materials. The introduction of SiC whiskers significantly improves high temperature creep resistance. Creep rates of material containing 20 vol.% SiC whiskers have been reported to be approximately two orders of magnitude lower than those of polycrystalline MoSi<sub>2</sub>. Alloying with WSi<sub>2</sub> has also been observed to improve creep rates, but to a lesser extent than SiC whisker additions. Effects of whisker additions and alloying have been seen to be additive. SiC whiskers have generally been observed to be more effective than SiC particles in terms of improving creep resistance, indicating an importance of reinforcement shape morphology on creep behavior of these composites.

An important observation which has been made is related to the competing effects of SiC reinforcing addition volume fraction and associated grain size of the MoSi<sub>2</sub> matrix on the creep resistance of SiC-MoSi<sub>2</sub> composites. As SiC is added, the grain size of the MoSi<sub>2</sub> is reduced. This leads to a maximum in the composite creep rate with volume fraction of SiC addition, as shown in Figure 7. Creep rates for 5, 10, and 20 vol.% SiC particle reinforced composites are higher than those of unreinforced MoSi<sub>2</sub>, while creep rates of a 40 vol.% SiC particle composite are substantially lower. This behavior is related to the fact that, with increasing SiC addition, the grain size of the MoSi<sub>2</sub> matrix is reduced. The reduced matrix grain size then promotes the operation of grain boundary sliding creep mechanisms.

The creep mechanisms in MoSi<sub>2</sub>-based composites appear to be a combination of dislocation glide/climb processes, as well as grain boundary sliding. The extent of grain

boundary sliding depends on the amount of silica phase present in the composite microstructure, as well as the grain size of the  $\text{MoSi}_2$  matrix. Wiederhorn et.al. [18] have shown that creep rates in tension are higher than creep rates in compression, due to the operation of these grain boundary sliding processes. Stress exponents for the creep of  $\text{MoSi}_2$  and many  $\text{MoSi}_2$  composites are in the vicinity of 2-5, while creep activation energies are in the range of 300-600 kJ/mole. The stress exponents suggest dislocation glide/climb processes and/or grain boundary sliding accommodated by dislocation plasticity. Although self-diffusion values for  $\text{MoSi}_2$  have not been extensively established, published values are 250 kJ/mole for Si in  $\text{MoSi}_2$  and 350-540 kJ/mole for Mo in  $\text{MoSi}_2$  [19]. These self-diffusion values are in the range of observed creep activation energies.

Figure 8 shows a comparison of  $\text{MoSi}_2$  composite creep rates to those of other metals and intermetallics [17]. It is clear that  $\text{MoSi}_2$  materials are significantly more creep resistant at elevated temperatures than these other materials. Although  $\text{MoSi}_2$ -based materials are not yet as creep resistant as silicon-based structural silicides such as  $\text{Si}_3\text{N}_4$  and  $\text{SiC}$ , with continued materials development, the creep resistance of these structural silicide materials may match or even surpass that of the structural ceramics.

#### **Crack Growth Behavior:**

The elevated temperature crack growth behavior of  $\text{MoSi}_2$  materials under static and fatigue conditions has been studied by Ramamurty et.al. [20,21]. Crack growth occurs by grain boundary controlled mechanisms. Crack growth behavior has been observed to be significantly affected by additions of carbon to the microstructure [21]. Crack growth rates at 1300 °C are reduced by approximately two orders of magnitude and the  $\Delta K$  for initial crack growth increased by a factor of two with the addition of carbon. This reflects a reduction in the amount of microstructural silica present, due to reactions between the silica phase and the carbon to form in-situ  $\text{SiC}$ . Figure 9 compares cyclic fatigue crack growth rates of  $\text{MoSi}_2$  materials to a number of other high temperature materials [20]. The  $\text{MoSi}_2$  materials compare quite favorably, both in terms of initial crack growth and the sensitivity of crack growth rate to the applied cyclic stresses.

**Fracture Toughness:**

Evans has nicely summarized the results of years of research on the toughening of structural ceramic materials [22]. The toughest ceramics have been composites incorporating continuous fiber reinforcements. The next toughest ceramics are those with ductile metal second phase dispersions. This is followed by ceramic materials incorporating zirconia transformation toughening effects. Second phase whiskers/particles/platelets fall next in the hierarchy of toughening mechanisms. Finally, matrix microcracking constitutes the least effective, although still significant, toughening approach.

In the work performed on MoSi<sub>2</sub> composites to date, the fracture toughness hierarchy which has been observed for ceramics has also been observed for MoSi<sub>2</sub> materials, as shown in Table 4. The toughest materials have incorporated refractory metal wires. These materials exhibit the "graceful failure" fracture morphology observed in continuous fiber ceramic matrix composites [23]. Composites containing Ta particles have shown the ductile phase toughening effects associated with the plastic deformation of ductile particles bridging the crack opening [24]. However, it is important to note that refractory metal reinforcements in MoSi<sub>2</sub> composites can exhibit significant reaction and oxidation problems.

ZrO<sub>2</sub> transformation toughening effects have been employed to toughen ZrO<sub>2</sub> particle-MoSi<sub>2</sub> matrix composites [25]. Figure 10 shows the fracture toughness of these composites as a function of the amount of Y<sub>2</sub>O<sub>3</sub> stabilizer in the ZrO<sub>2</sub> phase. Unstabilized ZrO<sub>2</sub> is observed to produce the maximum toughening effects in these types of composites. The unstabilized ZrO<sub>2</sub> tetragonal-to-monoclinic martensitic phase transformation occurs spontaneously upon cooling in the vicinity of 1100 °C. Thus, this transformation occurs above the brittle-to-ductile transition temperature of the MoSi<sub>2</sub> phase, where the MoSi<sub>2</sub> is still ductile. Since the ZrO<sub>2</sub> tetragonal-to-monoclinic phase transformation produces an approximately 4% expansional volumetric strain, this transformation effectively "pumps" dislocations into the MoSi<sub>2</sub> matrix, with potentially

beneficial effects on the brittle-to-ductile transition temperature of  $ZrO_2$ - $MoSi_2$  composites, as well as on improvements to the fracture toughness.

#### **SUMMARY:**

The above discourse has been an attempt to review the current salient features of the mechanical behavior of  $MoSi_2$  and  $MoSi_2$  composites. The mechanical behavior of  $MoSi_2$  single crystals was discussed, and possible and observed slip systems described. A most interesting feature of single crystal behavior is the extremely high strengths seen in the [001] orientation at elevated temperatures. The elevated temperature mechanical properties of polycrystalline  $MoSi_2$  improve with decreasing silica content and increasing grain size. The brittle-to-ductile transition temperature of polycrystalline  $MoSi_2$  can be reduced by the introduction of mobile dislocations as a result of high temperature prestraining. Transgranular fracture is the primary fracture mode of polycrystalline  $MoSi_2$  at room temperature. Carbon additions improve elevated temperature fracture toughness by removing deleterious silica phases.

Creep, crack growth, and fracture toughness can be substantially improved by the development of  $MoSi_2$ -based composites. An important feature associated with creep resistance is the balance between reinforcement strengthening and matrix grain size reduction due to the reinforcing phase. Creep mechanisms involve dislocation glide/climb and grain boundary sliding accommodated by plastic deformation. Good elevated temperature crack growth behavior is exhibited by  $MoSi_2$ -based composites, and is further improved by carbon additions. Toughening mechanisms for  $MoSi_2$ -based composites parallel those for structural ceramics. Continuous fiber reinforcement, ductile phase toughening, transformation toughening, second phase whiskers and particles, and matrix microcracking are all mechanisms operative in  $MoSi_2$ -based composites.

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**Table 1: Possible Slip Systems in MoSi<sub>2</sub> [5].**

<b>Slip System</b>	<b>Slip Plane Spacing d(nm)</b>	<b>Burgers Vector b(nm)</b>	<b>d/b ratio</b>
{013}<100>	0.202	0.3204	0.630
{010}<100>	0.160	0.3204	0.499
{110}<110>	0.226	0.4531	0.499
{110}1/2<111>	0.226	0.4531	0.499
{001}<100>	0.131	0.3204	0.409
{011}<100>	0.0989	0.3204	0.309
{110}1/2<331>	0.226	0.7848	0.288
{013}1/2<331>	0.202	0.7848	0.257
{011}1/2<111>	0.0989	0.4531	0.218

**Table 2: Slip systems observed in MoSi<sub>2</sub> single crystals [5,10].**

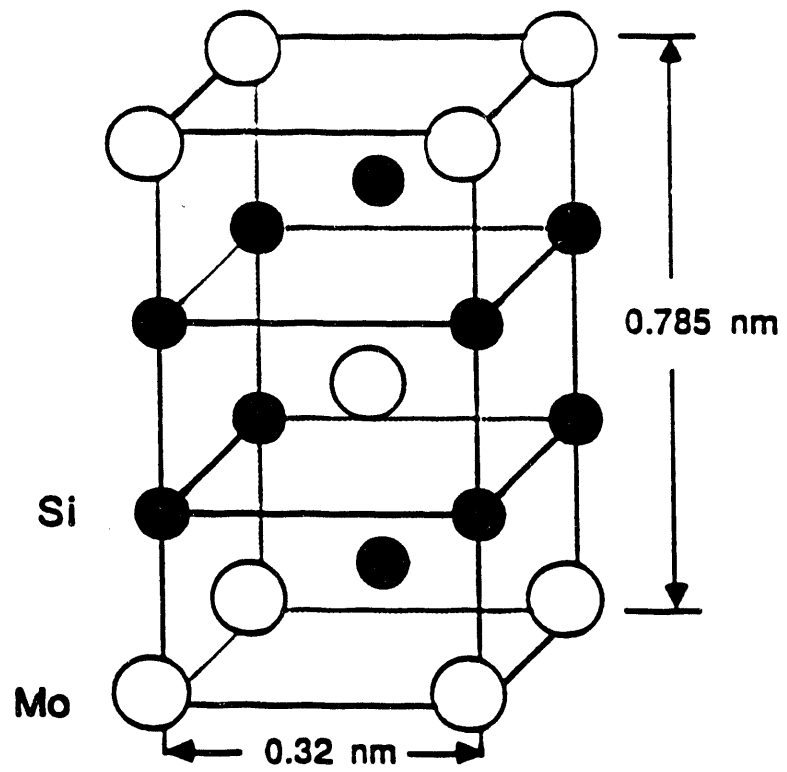
<b>Crystal Orientation</b>	<b>Temperature (°C)</b>	<b>Strain Rate (s<sup>-1</sup>)</b>	<b>Burgers Vector</b>	<b>Slip Plane</b>
[001]	900-1100	10 <sup>-5</sup>	1/2<331> 1/2<111> <110>	{013}
	1200-1400	10 <sup>-5</sup>	<100> <110> 1/2<111>	{001} {110}
	1400-1600	10 <sup>-4</sup>	<100> <110> 1/2<111>	{001} {110}
[021]	1000-1200	10 <sup>-4</sup> and 10 <sup>-5</sup>	1/2<111>	{110}
	1300-1400	10 <sup>-4</sup> and 10 <sup>-5</sup>	<100>	{001}
[771]	1000-1100	10 <sup>-4</sup>	1/2<331> 1/2<111> <110>	{013}
	1000	10 <sup>-5</sup>	<100>	{011}
	1100-1300	10 <sup>-5</sup>	<100>	{011}
	1200-1300	10 <sup>-4</sup>	<100>	{011}

**Table 3: Elastic stiffness and compliance constants for single crystal MoSi<sub>2</sub> [9].**

<b>Elastic Stiffness <math>C_{ij}</math></b>	<b>Elastic Compliance <math>S_{ij}</math></b>
$C_{11}$ 4.170	$S_{11}$ 2.611
$C_{33}$ 5.145	$S_{33}$ 2.051
$C_{44}$ 2.042	$S_{44}$ 4.897
$C_{66}$ 1.936	$S_{66}$ 5.165
$C_{12}$ 1.042	$S_{12}$ -0.586
$C_{13}$ 0.838	$S_{13}$ -0.330

**Table 4: Room temperature fracture toughness of MoSi<sub>2</sub>-Based Composites.**

<b>Type of Reinforcement</b>	<b>Highest Fracture Toughness (Mpa m<sup>1/2</sup>)</b>
Refractory metal (Nb, W, Mo) wires	Greater than 15
20 vol.% Ta particles	10
20 vol.% ZrO <sub>2</sub> particles	7.8
20 vol.% SiC whiskers	4.4
20 vol.% SiC particles	4.0
Polycrystalline MoSi <sub>2</sub>	3



**Figure 1: Crystal structure of MoSi<sub>2</sub>.**

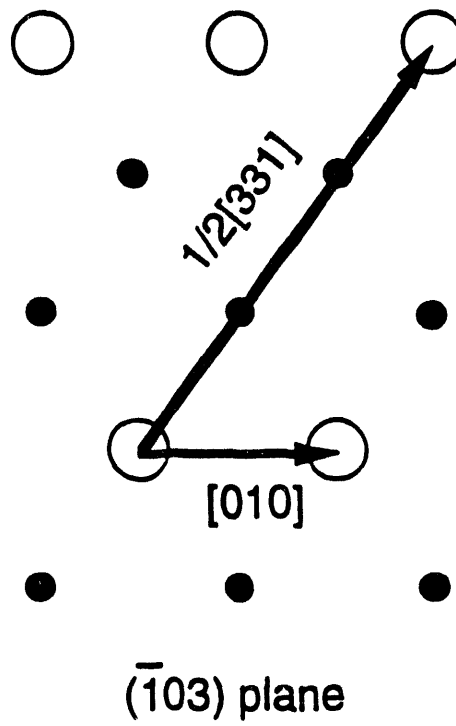
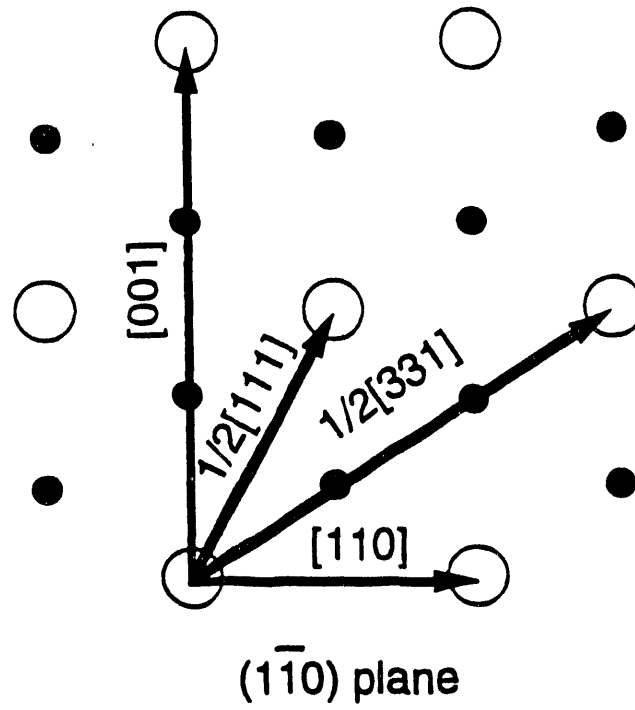


Figure 2: Important Burgers vectors for  $\text{MoSi}_2$  [10].

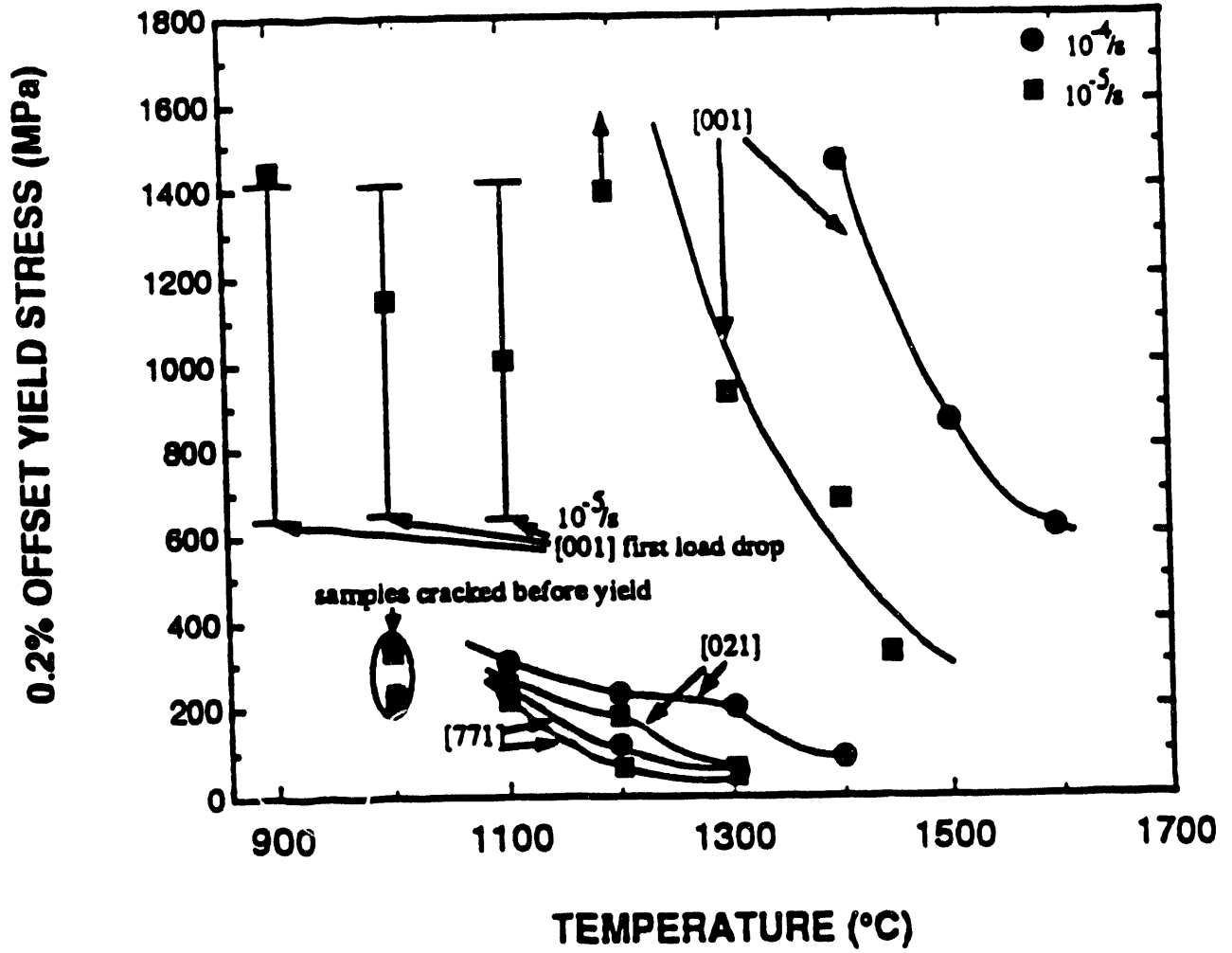


Figure 3: Yield stress of MoSi<sub>2</sub> single crystals as a function of temperature and crystallographic orientation [5,10].

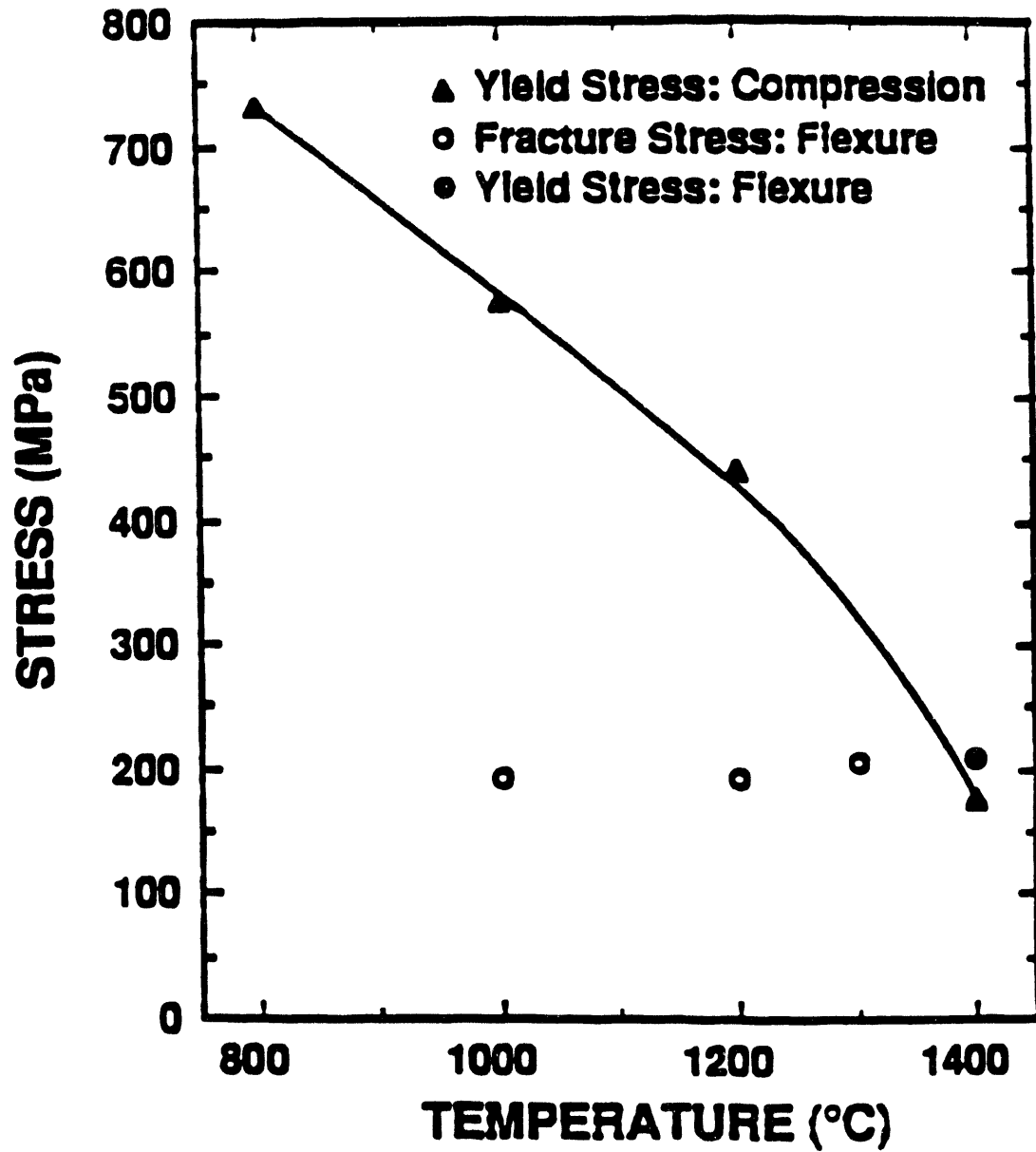


Figure 4: Yield stress versus temperature for low silica content, large grained polycrystalline MoSi<sub>2</sub> [11].

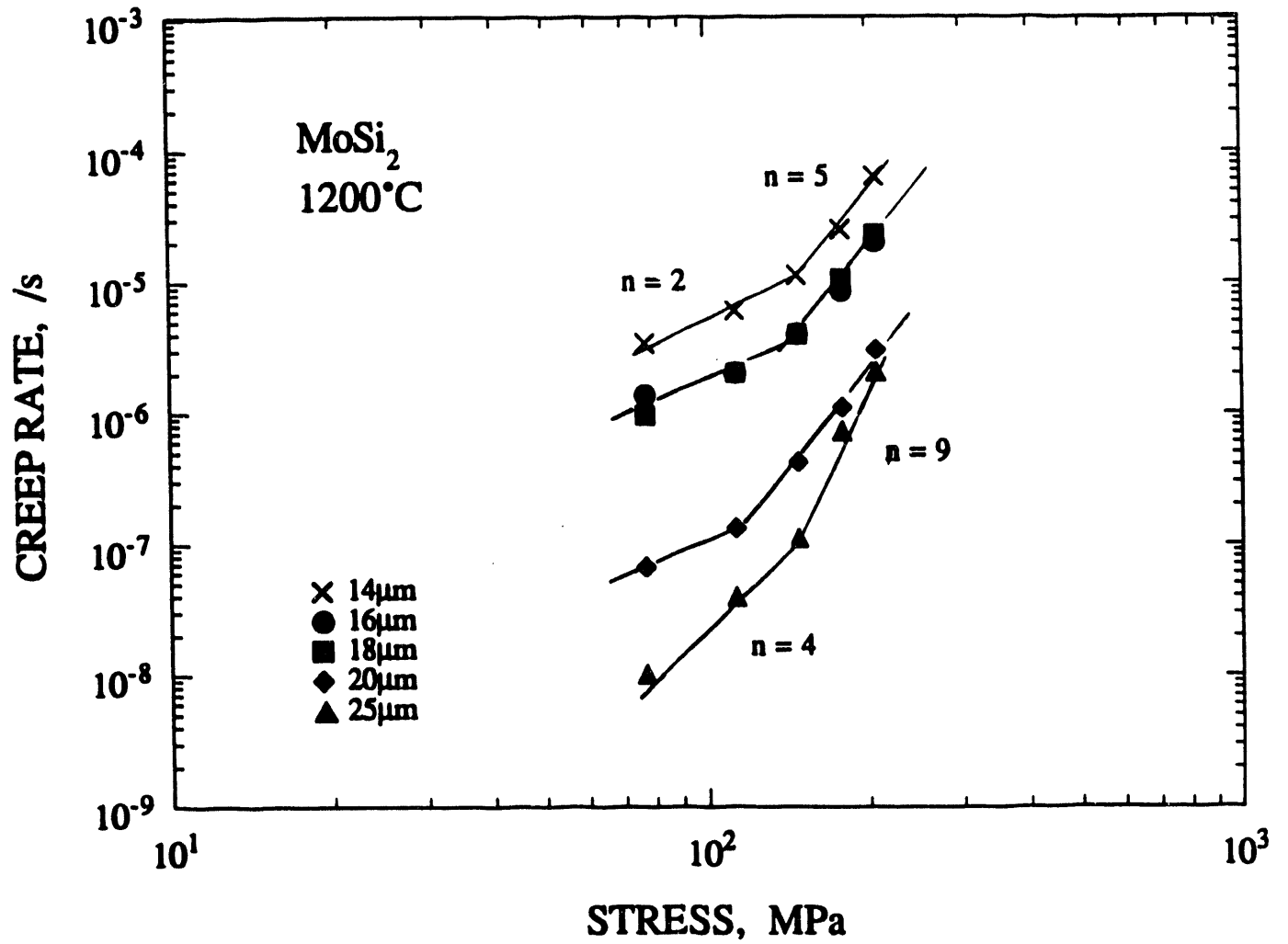
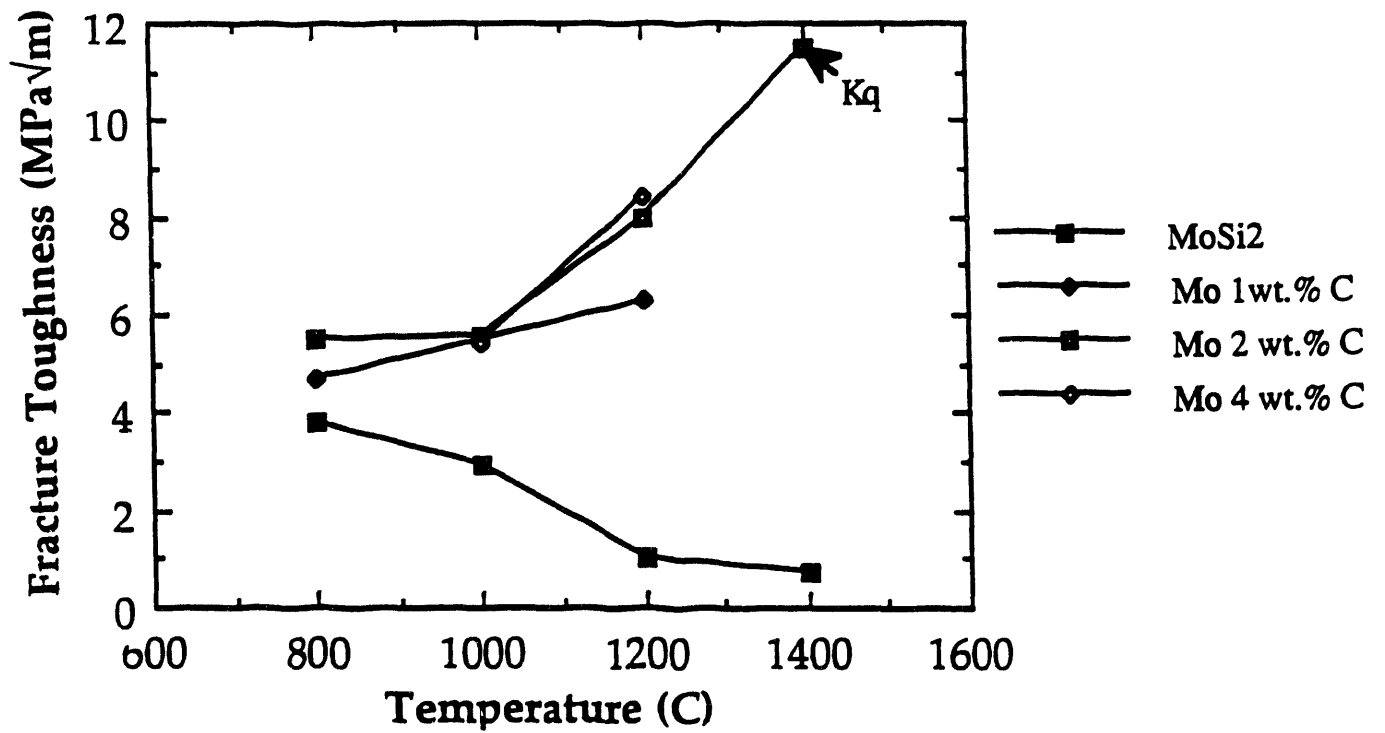


Figure 5: Creep rate of polycrystalline MoSi<sub>2</sub> as a function of grain size [13].

### Fracture Toughness with Additions of Carbon



**Figure 6: Effects of carbon additions on the elevated temperature fracture toughness of MoSi<sub>2</sub> [16].**

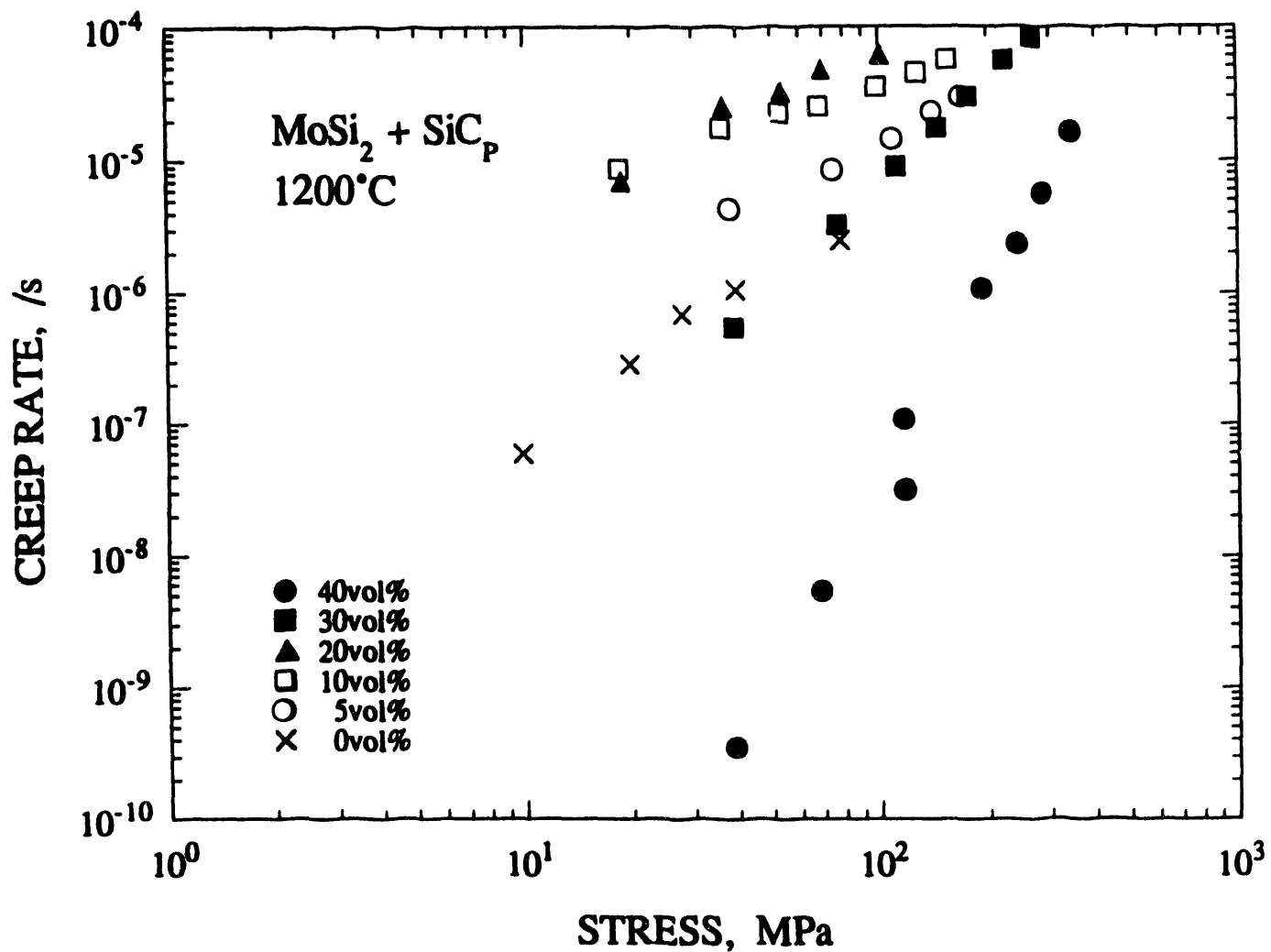
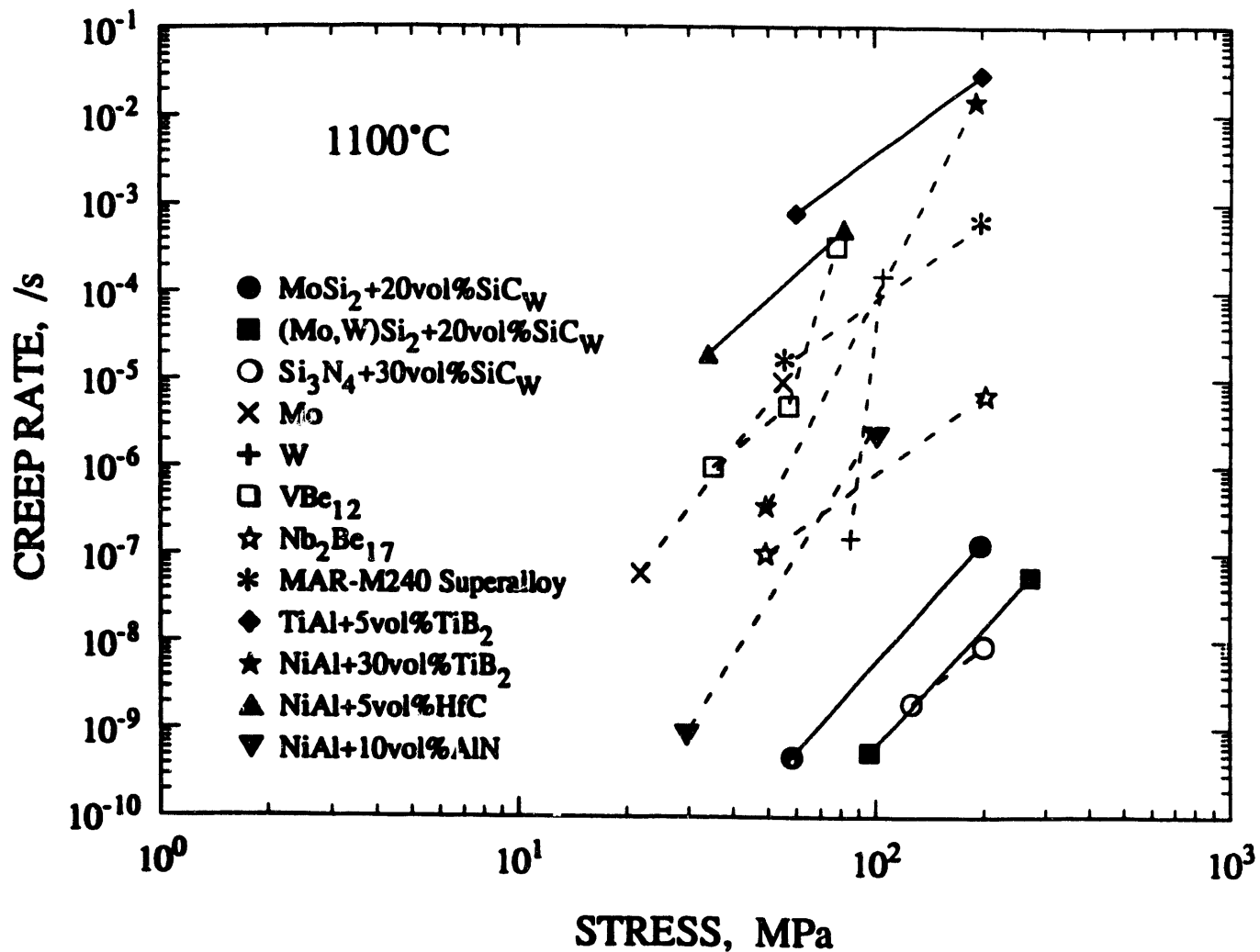


Figure 7: Creep rate of SiC particle-MoSi<sub>2</sub> matrix composites with different volume fractions of SiC phase [13].



**Figure 8: Comparison of the creep resistance of  $\text{MoSi}_2$ -based materials to other metals and intermetallic compounds [17].**

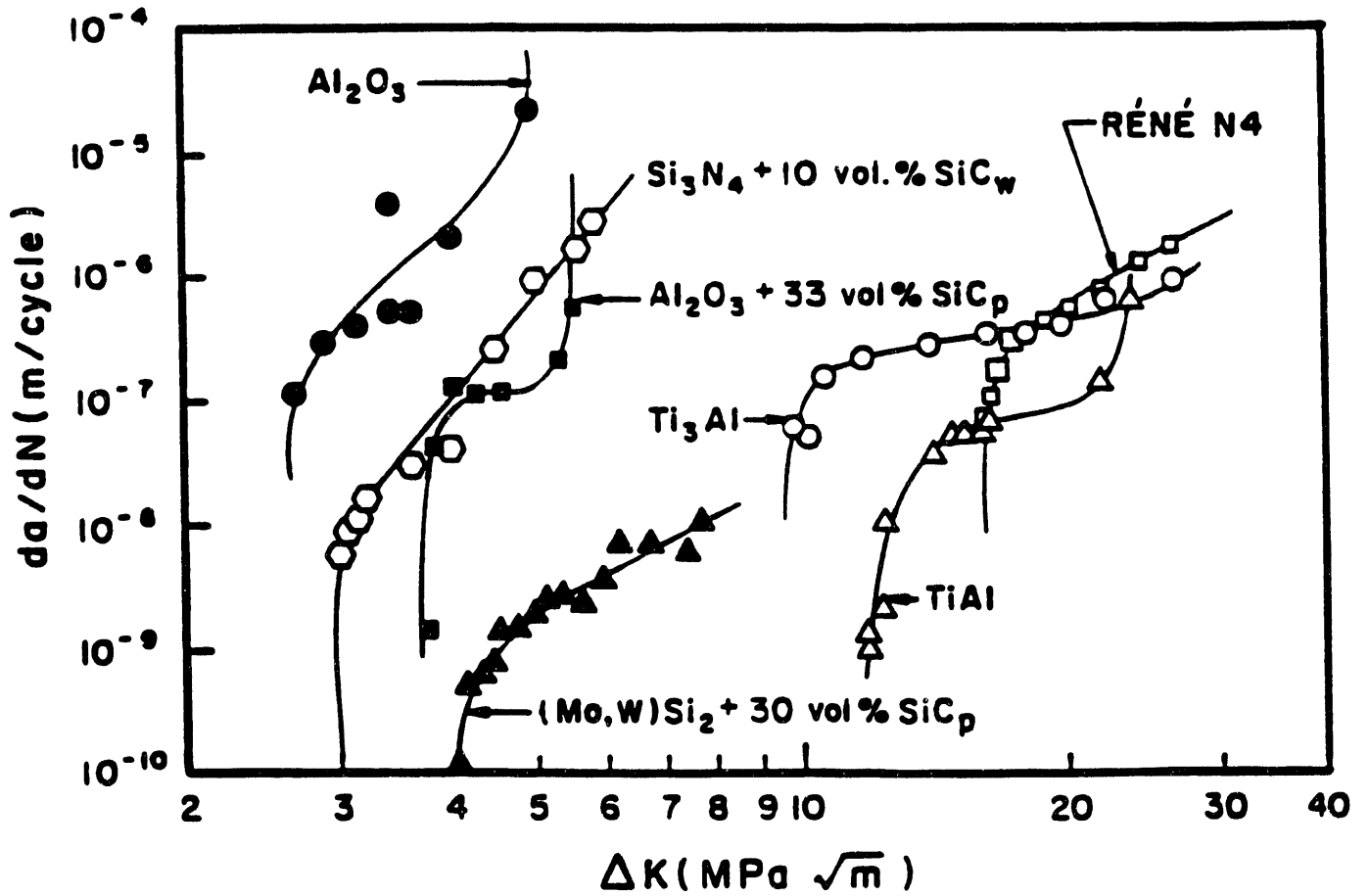


Figure 9: Comparison of high temperature cyclic crack growth of  $\text{MoSi}_2$  composites to that of other high temperature materials [20].

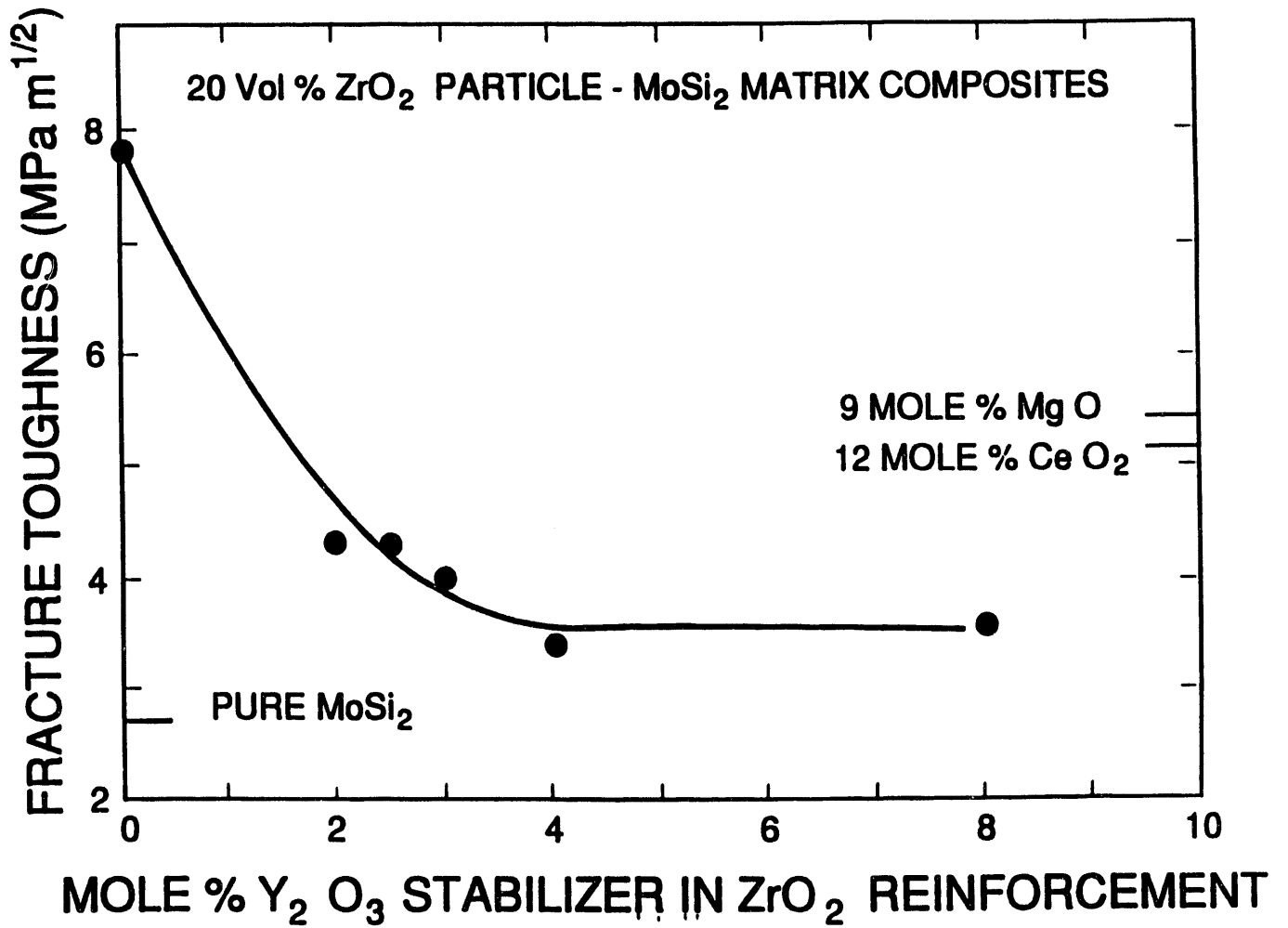


Figure 10: Fracture toughness of  $ZrO_2$  particle- $MoSi_2$  matrix composites as a function of  $ZrO_2$  stabilizer [25].

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