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ZrO_2 and ZrO_2/SiC Particle Reinforced-$MoSi_2$ Matrix Composites

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ABSTRACT

ZrO_2-$MoSi_2$ and ZrO_2/SiC-$MoSi_2$ composites were fabricated by hot pressing and hot pressing/HIP at 1700 °C. No reactions between ZrO_2, SiC, and $MoSi_2$ were observed. An amorphous silica glassy phase was present in all composites. Composites with unstabilized ZrO_2 particles exhibited the highest room temperature fracture toughness, reaching a level three times that of pure $MoSi_2$. Both the room temperature toughness and 1200 °C strength of ZrO_2/SiC-$MoSi_2$ composites were higher than ZrO_2-$MoSi_2$ composites, indicating beneficial effects of combined reinforcement phases. Low strength levels were observed at 1400 °C due to the presence of the silica glassy phase. Elimination of glassy phases and refinements in microstructural homogeneity are processing routes important to the optimization of the mechanical properties of these types of composites.

INTRODUCTION

Transformation toughening has been shown to be a very important toughening mechanism in zirconia-based materials [1-6]. In this mechanism, metastable tetragonal zirconia particles transform to monoclinic zirconia via a martensitic transformation, either spontaneously upon cooling or in the vicinity of crack tip stress fields. The volume change associated with the transformation produces the observed toughening effects. The transformation is sensitive to alloying additions to the ZrO_2 (the most important alloying species are MgO, CaO, CeO_2, and Y_2O_3), and particle size of the tetragonal phase.

Zirconia has also been added to alumina in order to increase the fracture toughness of ZrO_2-Al_2O_3 composites [7,8,9]. These composites are referred to as zirconia-toughened alumina, or ZTA. Some investigators have also added SiC whiskers to zirconia toughened materials in order to enhance high temperature mechanical properties [10,11].

Recent work has demonstrated that zirconia transformation toughening may also be employed to significantly toughen $MoSi_2$ based materials [12,13], when ZrO_2 particles are present as a dispersed phase in a $MoSi_2$ matrix. The chemical species ZrO_2 and $MoSi_2$ are stable with each other under inert conditions, and presence of the ZrO_2 does not significantly degrade composite oxidation resistance.

In the present investigation, we compare the fabrication, microstructures, and mechanical properties of ZrO_2 particle-$MoSi_2$ matrix and combined ZrO_2/SiC particle-$MoSi_2$ matrix composites.

EXPERIMENTAL

Fabrication:

ZrO_2-$MoSi_2$ Composites:

A range of commercial ZrO_2 powders from the Tosoh Corporation were employed. Powders containing 0, 2, 2.5, 3, 4, and 8 mole % Y_2O_3 were used. The 0 mole % Y_2O_3 material is unstabilized pure ZrO_2. The range of 2-4 mole % Y_2O_3 constitutes partially stabilized ZrO_2. The 8 mole % Y_2O_3 is fully stabilized ZrO_2. All Tosoh powders were high purity, with an average particle size of 0.3 micron.

$MoSi_2$ powder used was commercial powder from the Alfa Corporation. This powder was screened to -400 mesh prior to mixing with the various ZrO_2 powders. ZrO_2 and $MoSi_2$ powders were co-dispersed in an aqueous media at a pH of 2.5. Mechanical stirring and ultrasonification were employed. The solids loading was 60 wt.%. The powder co-dispersion was slip cast into a plaster of paris mold, and the slip cast body crushed into -100 mesh feed powder for hot pressing. Hot pressing consolidation of composites was performed at 1700 ^oC and 32 MPa pressure, using grafoil-lined graphite dies and an argon atmosphere. Hot pressed composites were 94-95% dense, and contained 20 vol.% ZrO_2 particles.

ZrO_2/SiC-$MoSi_2$ Composites:

Tosoh zirconia powders containing 0, 2.5, and 8 mole % Y_2O_3 were employed. Alpha SiC (0.5 micron particle size) SiC powder from the H.C. Starck Corporation was used. The $MoSi_2$ powder was H.C. Starck Grade C, with a particle size of 0.9-1.3 micron, and an indicated oxygen content of 2 wt.%.

ZrO_2, SiC, and $MoSi_2$ powders were roll-blended dry with WC balls. The blend was then dry milled using a Megapact vibratory mill with WC balls. The powder was contained in a polyethylene jar, and milling time was one hour.

Milled powders were hot pressed at 1700 °C and 32 MPa pressure for 30 minutes, using grafoil-lined graphite dies and an argon atmosphere. Hot pressed composites were then hot isostatically pressed (HIPed) in a containerless manner at 1700 °C and 207 MPa for 30 minutes in argon. HIPed composites were 100% dense, and contained 10 vol.% ZrO_2 and 10 vol.% SiC particles.

Microstructures:

Composite microstructures were examined with light optical, XRD, SEM, and TEM techniques. Light optical and SEM techniques were employed to assess microstructural homogeneity. X-ray diffraction was performed to determine phases present after fabrication. Transmission electron microscopy was used to describe composite substructures. TEM studies of substructures were performed using a Philips CM30 TEM at 300 kV.

Mechanical Properties:

Microhardness indentation techniques were employed to determine the room temperature fracture toughness of the composites. Vickers indentations were used and the approach of Anstis et. al. [14] was adopted to calculate fracture toughness values.

Four-point bend strength was measured as a function of temperature. Bend specimen dimensions were 2.54 x 5.08 x 25.4 mm. Four-point bend test fixtures had an outer span of 19 mm and an inner span of 9.5 mm. A hardened tool steel self-aligning bend fixture was employed at room temperature. Elevated temperature bend tests were conducted using a ceramic bend fixture, with the specimens heated with a $MoSi_2$ element mechanical testing furnace. All mechanical tests were performed on an Instron test frame.

RESULTS

Microstructures:

Representative SEM micrographs of the polished microstructures of 20 vol.% ZrO_2-$MoSi_2$ and 10 vol.% ZrO_2/10 vol.% SiC-$MoSi_2$ composites are shown in Figure 1. As may be seen, the ZrO_2/SiC-$MoSi_2$ composites exhibited significantly more homogeneous microstructures than the ZrO_2-$MoSi_2$ composites. This difference is due to the difference in fabrication procedures for the two types of composites. It suggests that high energy vibratory milling followed by hot pressing/HIP consolidation is a preferred fabrication methodology to aqueous co-dispersion of powders with only hot pressing consolidation. For the case of the ZrO_2-$MoSi_2$ composites, the ZrO_2 particles tended to be rather inhomogeneously clustered between regions of $MoSi_2$ phase, rather than discretely dispersed as individual particles. For the ZrO_2/SiC-$MoSi_2$ composites, the extent of clustering was reduced, though not totally eliminated.

X-Ray Diffraction Phase Analysis:

X-ray diffraction studies of the ZrO_2-$MoSi_2$ composites after fabrication consolidation at 1700 ^{o}C indicated the presence of only ZrO_2 and $MoSi_2$ phases. The crystal structure of the ZrO_2 phase was dependent on the amount of Y_2O_3 addition. The 0YZrO_2 was monoclinic, the 2-4YZrO_2 was tetragonal, while the 8YZrO_2 was cubic. The $MoSi_2$ exhibited the tetragonal crystal structure.

Similarly, for the ZrO_2/SiC-$MoSi_2$ composites, only the ZrO_2, SiC, and $MoSi_2$ phases were observed. ZrO_2 crystal structures were the same as in the ZrO_2-$MoSi_2$ composites. The SiC exhibited an alpha crystal structure, while the $MoSi_2$ phase was again tetragonal.

These phase analysis results indicate that there was no reaction between the species ZrO_2, SiC, and $MoSi_2$ at the temperature of 1700 ^{o}C employed for composite consolidation.

Substructures:

TEM substructures of ZrO_2-$MoSi_2$ and ZrO_2/SiC-$MoSi_2$ composites containing 0YZrO_2 and 2.5YZrO_2 compositions are shown in Figures 2 and 3. A prominent amorphous glassy silica phase was observed in all composites. This glassy phase was most prominent in the areas of ZrO_2 and ZrO_2/SiC particle clusters. It is also highly likely that $MoSi_2$ grain boundaries contained a thin grain boundary silica phase. A qualitative TEM chemical analysis of the glassy phase showed it to be essentially SiO_2, with a small amount of Zr, Y, and Mo present. Thus, the glassy phase displayed dissolved elements from some of the composite constituent phases.

In Figure 2, the 20 vol.% 0YZrO_2-$MoSi_2$ composite exhibited ZrO_2 particles that were monoclinic. Thus, the tetragonal-to-monoclinic phase transformation occurred spontaneously upon cooling from the composite fabrication temperature. The $MoSi_2$ phase in this composite showed a high density of both dislocations and microcracks, which were induced by the spontaneous zirconia transformation [13]. Densities of $MoSi_2$ phase dislocations and microcracks were lower in the 10 vol.% 0YZrO_2/SiC-$MoSi_2$ composite. This is likely due to the lower volume fraction of zirconia in this composite.

In Figure 3, the 20 vol.% 2.5YZrO_2-$MoSi_2$ composite showed ZrO_2 particles which were tetragonal. In this material, the tetragonal-to-monoclinic phase transformation had not occurred spontaneously upon cooling. Dislocation densities and microcracking were observed to be low in the $MoSi_2$ phase [13]. The substructure of the 10 vol.% 2.5YZrO_2/SiC-$MoSi_2$

composite was similar to the ZrO_2-$MoSi_2$ composite, except for the presence of the fine SiC particles.

Room Temperature Fracture Toughness:

The room temperature indentation fracture toughness of 20 vol.% ZrO_2-$MoSi_2$ composites containing various mole % levels of Y_2O_3 stabilizer in the ZrO_2 reinforcement is shown in Figure 4. A similar plot for zirconia-toughened Al_2O_3 (ZTA) is also shown [7], for comparison to zirconia-toughened $MoSi_2$ (ZTM).

As may be seen, the highest toughness for the ZrO_2-$MoSi_2$ composites occured with unstabilized (0 mole % Y_2O_3) ZrO_2. In this case the composite toughness was three times higher than that of pure $MoSi_2$, a substantial toughening effect. Partially stabilized (2-4 mole % Y_2O_3) and fully stabilized (8 mole % Y_2O_3) compositions showed lower toughness levels, but still above pure $MoSi_2$. By way of comparison, the toughness of unstabilized ZTA is only slightly above pure Al_2O_3, with maximum toughening occurring in the partially stabilized zirconia range. The toughness of fully stabilized ZTA is reported lower than that of pure Al_2O_3.

Figure 5 shows effects of volume fraction ZrO_2 reinforcement on the room temperature fracture toughness of 2.5YZrO_2-$MoSi_2$ ZTM composites. Also shown for comparison are volume fraction effects in 2.0YZrO_2-Al_2O_3 ZTA composites. The ZTM composites exhibited a linear relationship between fracture toughness and volume fraction ZrO_2 reinforcement. For the ZTA composites, the behavior roughly parallels the ZTM composites up to 45 vol.%, then deviates toward the somewhat lower value of the 100% 2.0YZrO_2.

A comparison of indentation fracture toughness as a function of crack length (basically, measured indentation toughness as a function of indentation load at 20, 30, and 50 kg) for ZrO_2-$MoSi_2$ composites and ZrO_2/SiC-$MoSi_2$ composites is shown in Figure 6. The slightly lower absolute toughness values for the 20 vol.% 0YZrO_2-$MoSi_2$ material in comparison to the value in Figure 4 are attributed to operator judgment in measuring the length of indentation cracks (particularly when significant microcracking is present), since different investigators made these two sets of measurements.

The most striking aspect in Figure 6 is the fact that the fracture toughness of the 0YZrO$_2$/SiC-MoSi$_2$ composite was higher than the toughness of the 0YZrO$_2$-MoSi$_2$ composite, this despite the fact that the former material contained only 10 vol.% ZrO$_2$ while the latter contained 20 vol.% ZrO$_2$. Toughness values for the 2.5YZrO$_2$/SiC composite were slightly higher than for the 2.5YZrO$_2$ composite, while 8YZrO$_2$ composite toughnesses were roughly comparable. All composites exhibited a trend of increasing toughness with increasing indentation crack length (increasing indentation load), suggesting R-curve type behavior in these materials.

Bend Strength versus Temperature:

Bend tests were performed at room temperature, 1200 ^{o}C, and 1400 ^{o}C in air. Figure 7 plots strength versus temperature for the ZrO$_2$-MoSi$_2$ and ZrO$_2$/SiC-MoSi$_2$ composites. Room temperature strengths are ultimate brittle fracture strengths. Strengths at 1200 ^{o}C and 1400 ^{o}C are yield strengths in bending, since composites exhibited plastic deformation at elevated temperatures. Yield strength in bending was defined as the strength level at 0.2% plastic bending strain.

At room temperature, composites containing 2.5YZrO$_2$ showed the highest strength levels, while composites containing 0YZrO$_2$ exhibited the lowest strengths. The 8YZrO$_2$ composites were intermediate. At 1200 ^{o}C, strengths of the ZrO$_2$/SiC-MoSi$_2$ composites were roughly double those of the ZrO$_2$-MoSi$_2$ composites, for all ZrO$_2$ compositions. Thus, the presence of the SiC phase produced a significant strengthening effect at 1200 ^{o}C. At 1400 ^{o}C, the strength of all composites decreased dramatically to a low level. This decrease is attributed to the presence of the glassy silica phase in the composites.

DISCUSSION

ZrO_2 Transformation Toughening Effects:

The present results indicate that ZrO_2 produces substantial room temperature transformation toughening effects in $MoSi_2$ based (ZTM) composites. Toughening effects are most pronounced for unstabilized (0 mole % Y_2O_3) ZrO_2, in distinct contrast to the behavior of ZTA. In the case of unstabilized ZTA, toughening appears to be associated with microcracking mechanisms [9]. A very intriguing additional aspect occurring in unstabilized ZrO_2-$MoSi_2$ composites is the "pumping" of dislocations into the $MoSi_2$ matrix as a result of the spontaneous ZrO_2 tetragonal-to-monoclinic phase transformation. Upon cooling from the fabrication temperature (1700 °C), this transformation initiates in the vicinity of 1175 °C [6]. The unstabilized zirconia transformation temperature is above the brittle-to-ductile transition of $MoSi_2$, and so dislocation "pumping" occurs as a result of the spontaneous ZrO_2 transformation strains. No such phenomena occurs in the ZTA composite system, since Al_2O_3 possesses insufficient dislocation plasticity at the unstabilized ZrO_2 transformation temperature [9]. It is possible that the increased dislocation density induced in the $MoSi_2$ matrix by the unstabilized ZrO_2 may be a contributing factor to the observed toughening effects. For such a mechanism to operate, these induced dislocations would have to be mobile at the $MoSi_2$ crack tip at room temperature, well below the brittle-to-ductile transition. Clearly, additional investigations targeted at definatively establishing (or ruling out) such a toughening mechanism are warranted.

Present results indicate that crack-tip-induced transformation toughening effects were not yet optimized in current partially stabilized (2-4 mole % Y_2O_3) ZrO_2 ZTM composites. This is in contrast to ZTA composites, where maximum toughening effects occur [7,9]. It is believed that the lack of sufficient microstructural dispersion of individual partially stabilized

ZrO_2 particles in the $MoSi_2$ matrix of the current composites is responsible for repressing these crack tip transformation effects. A recent investigation has demonstrated that partially stabilized ZTM composites exhibit R-curve behavior, and that such behavior is controlled by the composite critical transformation stress [15]. A high critical stress level observed in current ZTM composites is considered indicative of the inhibition of crack-tip transformation toughening effects due to microstructural inhomogeneity.

Effects of Combined ZrO_2 and SiC Reinforcements:

ZrO_2/SiC-$MoSi_2$ composites were stronger than ZrO_2-$MoSi_2$ composites at 1200 °C, and were also tougher materials at room temperature. The strengthening observed at 1200 °C is possibly related to the presence of the SiC particulate phase more effectively impeding dislocation motion at this temperature. Significant improvements in elevated temperature strength with the presence of a SiC reinforcing phase have certainly been observed in previous work [16]. Present results might suggest that SiC is a more effective elevated temperature strengthening phase than ZrO_2. However, the dispersion homogeneity of the ZrO_2 phase in the ZrO_2-$MoSi_2$ composites was certainly not optimum, and the dispersion of reinforcing particulate phases was clearly significantly improved in the ZrO_2/SiC-$MoSi_2$ composites. It is possible that once a microstructural condition of isolated, submicron ZrO_2 particles in the $MoSi_2$ matrix is achieved, ZrO_2 particles may prove to be as effective obstacles to dislocation motion as SiC particles.

The room temperature fracture toughness levels of unstabilized 0YZrO_2/SiC-$MoSi_2$ and partially stabilized 2.5YZrO_2/SiC-$MoSi_2$ composites were higher than their corresponding ZrO_2-$MoSi_2$ composite counterparts, despite the fact that these composites contained half the amount of ZrO_2 phase. It is believed that some of this improvement is related to the improved dispersion of ZrO_2 particles achieved in the ZrO_2/SiC-$MoSi_2$ composites in comparison to the ZrO_2-$MoSi_2$ composites. However, recent work [17] has shown that a dispersed particulate

SiC phase can produce room temperature toughening effects of itself. Thus, in ZrO_2/SiC-$MoSi_2$ composites, it appears that ZrO_2 particle and SiC particle toughening effects are certainly at least additive, and may be interactive in some way. One possible interaction mechanism might be related to the thermal expansion mismatch stresses associated with SiC particles enhancing ZrO_2 transformation toughening effects.

Effects of Glassy Phase on Elevated Temperature Strength:

Present results clearly demonstrate the detrimental effects of the presence of a microstructural glassy phase on the elevated temperature mechanical properties of both ZrO_2-$MoSi_2$ and ZrO_2/SiC-$MoSi_2$ composites. Such detrimental glassy phase effects have also been observed in polycrystalline $MoSi_2$ [18]. When a glassy phase is present along the grain boundaries of the $MoSi_2$ matrix, it acts to induce grain boundary sliding in preference to dislocation motion as the dominant deformation mechanism. In the presence of the glassy phase, grain boundary sliding becomes more dominant with increasing temperature, as the viscosity of the glassy phase decreases. In the present composites, dislocation motion was the likely primary deformation mechanism at 1200 ^oC, since the grain boundary glassy phase viscosity was high at this temperature. However, at 1400 ^oC the glassy phase viscosity was sufficiently low that grain boundary sliding likely became the dominant deformation mechanism, with associated macroscopic plastic strain occurring at low stress levels. This is the probable reason for the significant reduction in composite strength observed at 1400 ^oC.

Aspects for Composite Mechanical Property Optimization:

There are two clear avenues to follow in order to optimize both the high and low temperature mechanical properties of ZrO_2-$MoSi_2$ and ZrO_2/SiC-$MoSi_2$ composites. The first is elimination of the silica glassy phase and the second is refinement in the microstructural dispersion of reinforcing particulate phases.

The glassy silica phase occurs in $MoSi_2$ composites since it is present on the surfaces of the starting commercial $MoSi_2$ powders used to fabricate these composites. Recent work has demonstrated that carbon additions to $MoSi_2$ are very effective in eliminating the silica glassy phase, via reactions between carbon and silica at elevated fabrication temperatures to form SiC and gaseous CO [18]. Removal of the glassy phase markedly improved the high temperature mechanical properties of polycrystalline $MoSi_2$. Similar improvements with carbon additions are to be expected for the present composites.

The second aspect involves developing fabrication techniques designed to produce a microstructure of isolated, well-dispersed, submicron ZrO_2 and SiC particles in the $MoSi_2$ matrix. This suggests the use and optimization of high energy powder milling techniques such as vibratory milling and mechanical alloying to intimately blend composite constituent powders, and hot isostatic pressing (HIP) to consolidate these powders at lower temperatures where microstructural coarsening during the consolidation process may be minimized. Improvements in microstructural homogeneity were clearly achieved in present ZrO_2/SiC-$MoSi_2$ composites when vibratory milling and HIP consolidation were introduced.

CONCLUSIONS

ZrO_2-$MoSi_2$ and ZrO_2/SiC-$MoSi_2$ composites were fabricated by hot pressing and hot pressing/hot isostatic pressing at 1700 ^{o}C, and their microstructures and mechanical properties evaluated. Under the fabrication conditions, no reactions between ZrO_2, SiC, and $MoSi_2$ were observed. A combination of powder vibratory milling followed by hot pressing/HIP consolidation was observed to improve the microstructural homogeneity of ZrO_2/SiC-$MoSi_2$ composites, as compared to ZrO_2-$MoSi_2$ composites which were fabricated by aqueous co-dispersion of powders followed by hot pressing. An amorphous glassy silica phase was observed in all composite materials, due to silica present on the surfaces of the starting commercial $MoSi_2$ powders.

The presence of an unstabilized ZrO_2 particulate phase in ZrO_2-$MoSi_2$ (ZTM) composites increased the room temperature indentation fracture toughness by a factor of three over that of pure $MoSi_2$. This is in contrast to the behavior of ZrO_2-Al_2O_3 (ZTA) composites. For unstabilized ZrO_2, microcracking and dislocation "pumping" were seen in the $MoSi_2$ matrix. Observed composite toughness levels for partially stabilized and fully stabilized ZrO_2 were lower than those of unstabilized ZrO_2. Microstructural inhomogeneity of the partially stabilized ZrO_2 particles in the $MoSi_2$ matrix was considered deleterious to the optimum occurrence of crack-tip-induced transformation toughening effects. Fracture toughness of partially stabilized ZrO_2-$MoSi_2$ composites increased linearly with increasing ZrO_2 volume fraction. The fracture toughness of unstabilized and partially stabilized ZrO_2/SiC-$MoSi_2$ composites was greater than than that of ZrO_2-$MoSi_2$ composites, and was likely due to a combination of microstructural homogeneity and possible ZrO_2-SiC interactive effects.

ZrO_2/SiC-$MoSi_2$ composites were stronger than ZrO_2-$MoSi_2$ composites at 1200 ^oC. This may be primarily due to the improved microstructural homogeneity of the former composites as compared to the latter, although a possible greater effectiveness of SiC particles over ZrO_2 particles to retard elevated temperature dislocation motion in the $MoSi_2$ matrix cannot be discounted. The strength of all composites was degraded at 1400 ^oC due to the presence of an amorphous glassy silica phase in the $MoSi_2$ matrix. This glassy phase induced grain boundary sliding to occur at low stress levels at 1400 ^oC, rather than the operation of dislocation plasticity taking place at higher stress levels.

The results of this investigation clearly define two processing routes for the optimization of both the high and low temperature mechanical properties of composites of this type. The first is to eliminate the silica glassy phase, and the second is to refine the microstructural dispersion of the reinforcing particulate phases.

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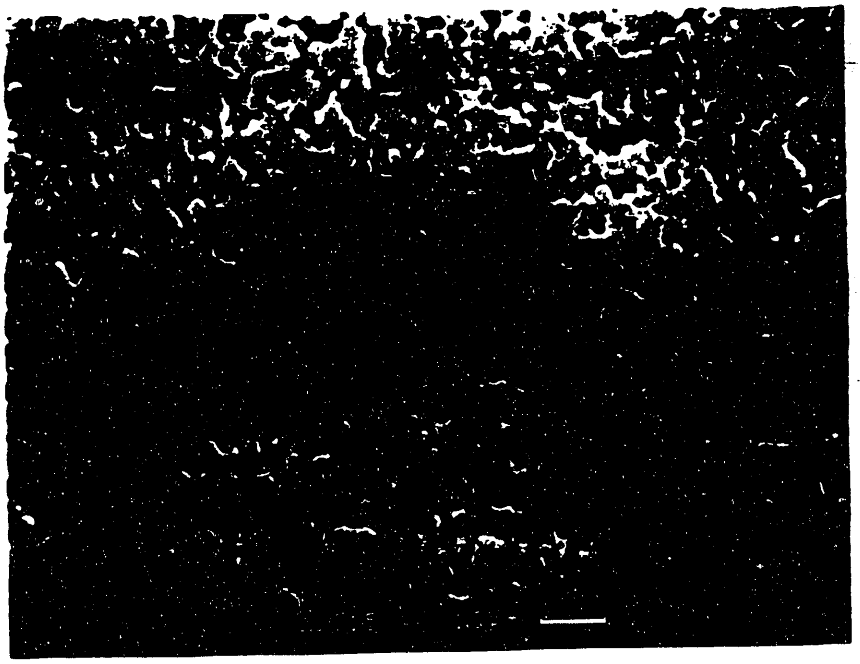
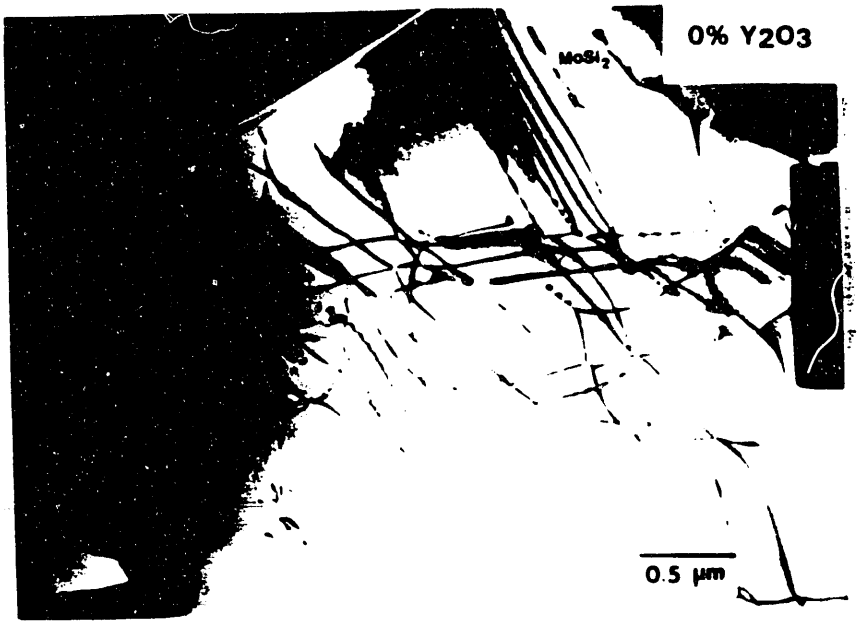
20 vol.% ZrO_2-$MoSi_2$

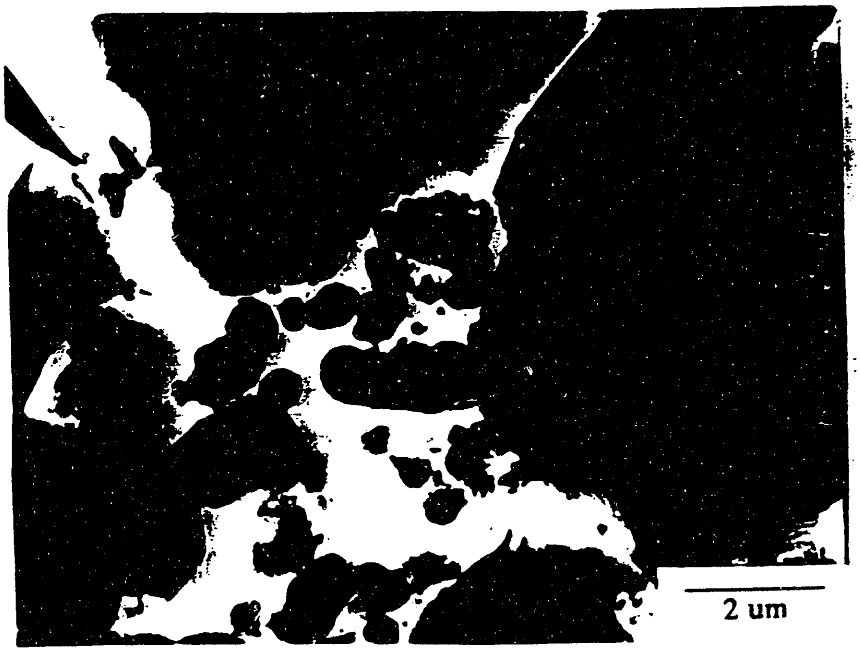


10 vol.% ZrO_2/10 vol.% SiC-$MoSi_2$

Figure 1: Comparison of the polished microstructures of 20 vol.% ZrO_2-$MoSi_2$ and 10 vol.% ZrO_2/10 vol.% SiC-$MoSi_2$ composites.

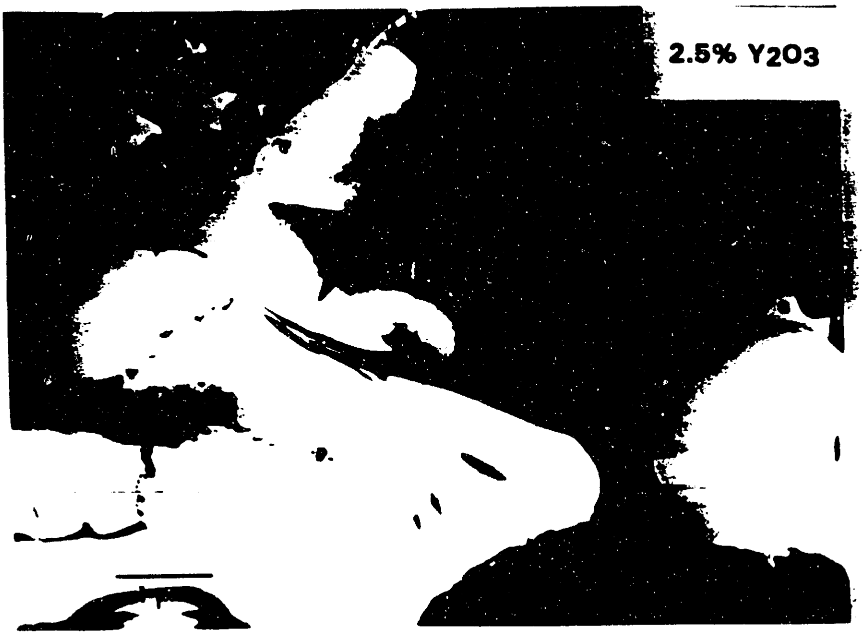


20 vol.% 0YZrO$_2$-MoSi$_2$



10 vol.% 0YZrO$_2$/10 vol.% SiC-MoSi$_2$

Figure 2: TEM substructures of ZrO$_2$-MoSi$_2$ and ZrO$_2$/SiC-MoSi$_2$ composites containing the 0YZrO$_2$ composition.



20 vol.% 2.5YZrO_2-$MoSi_2$

10 vol.% 2.5YZrO_2/10 vol.% SiC-$MoSi_2$

Figure 3: TEM substructures of ZrO_2-$MoSi_2$ and ZrO_2/SiC-$MoSi_2$ composites containing the 2.5YZrO_2 composition.

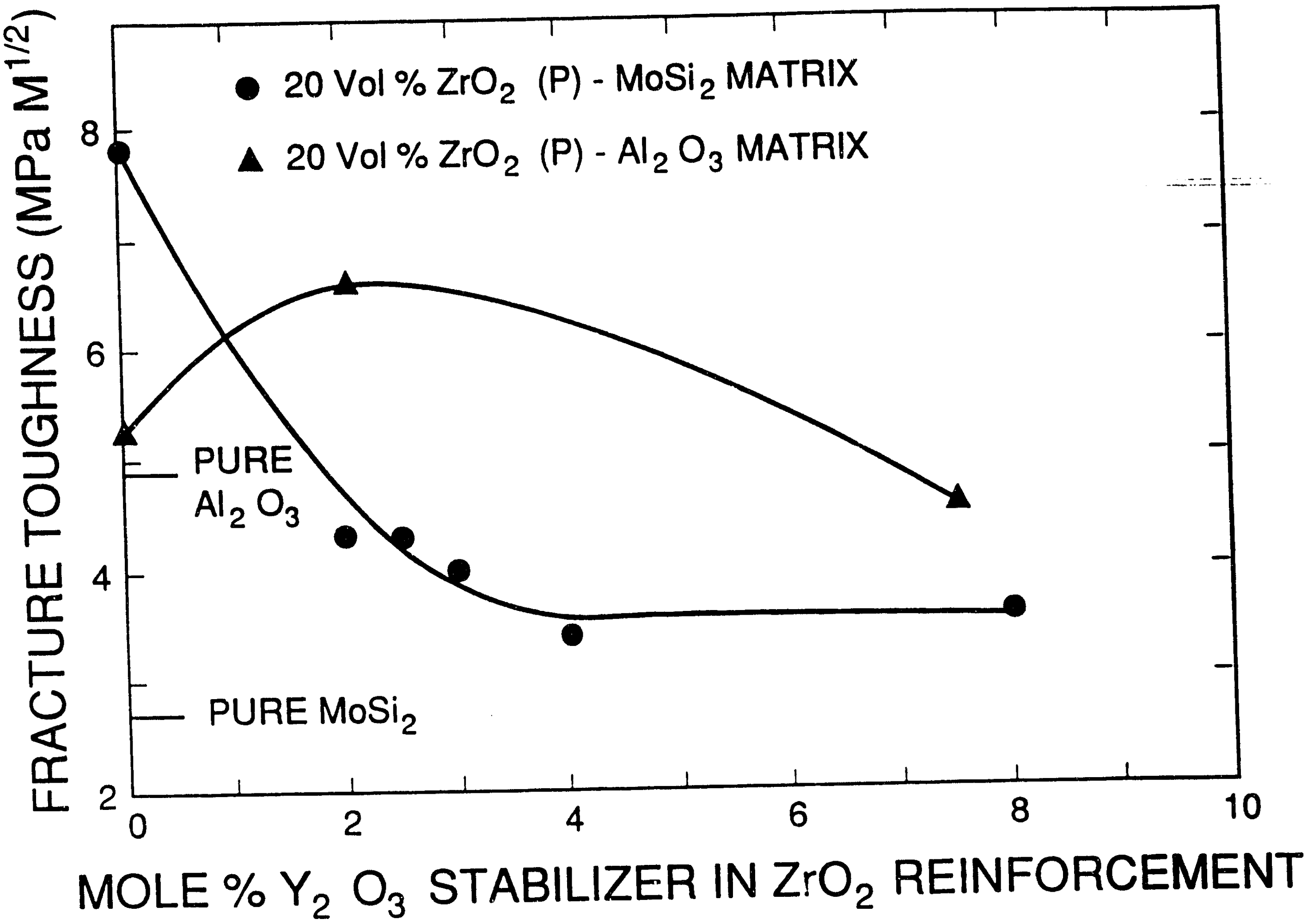


Figure 4: Room temperature fracture toughness of 20 vol.% ZrO_2-$MoSi_2$ composites as a function of mole % Y_2O_3 stabilizer in the ZrO_2 reinforcement. Comparison to the behavior of 20 vol.% ZrO_2-Al_2O_3 composites.

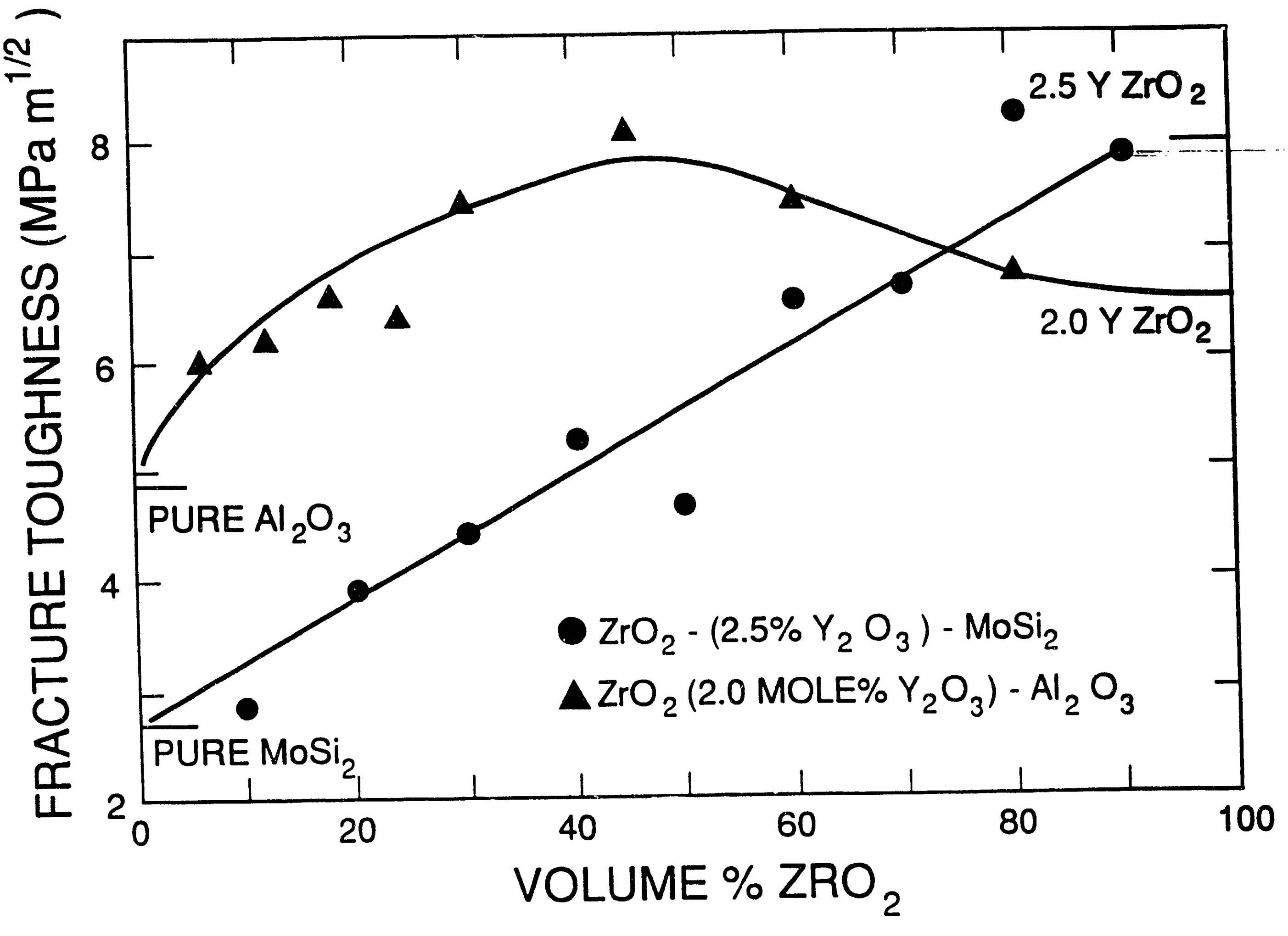


Figure 5: Effects of volume fraction ZrO_2 reinforcement on the room temperature fracture toughness of 2.5YZrO_2 particle-$MoSi_2$ matrix composites.

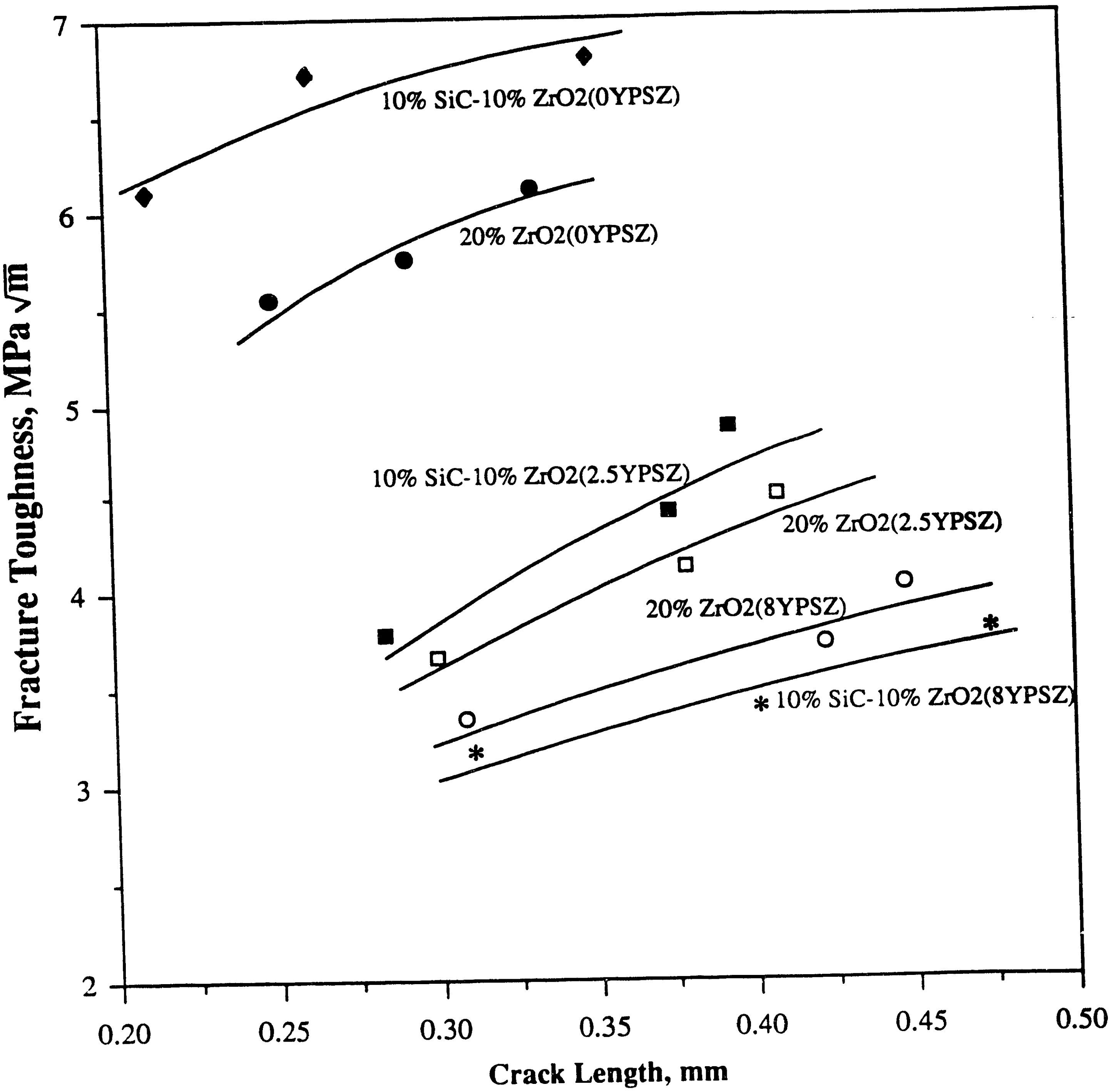


Figure 6: Comparison of the indentation fracture toughness of ZrO_2-$MoSi_2$ and ZrO_2/SiC-$MoSi_2$ composites, as a function of indentation crack length.

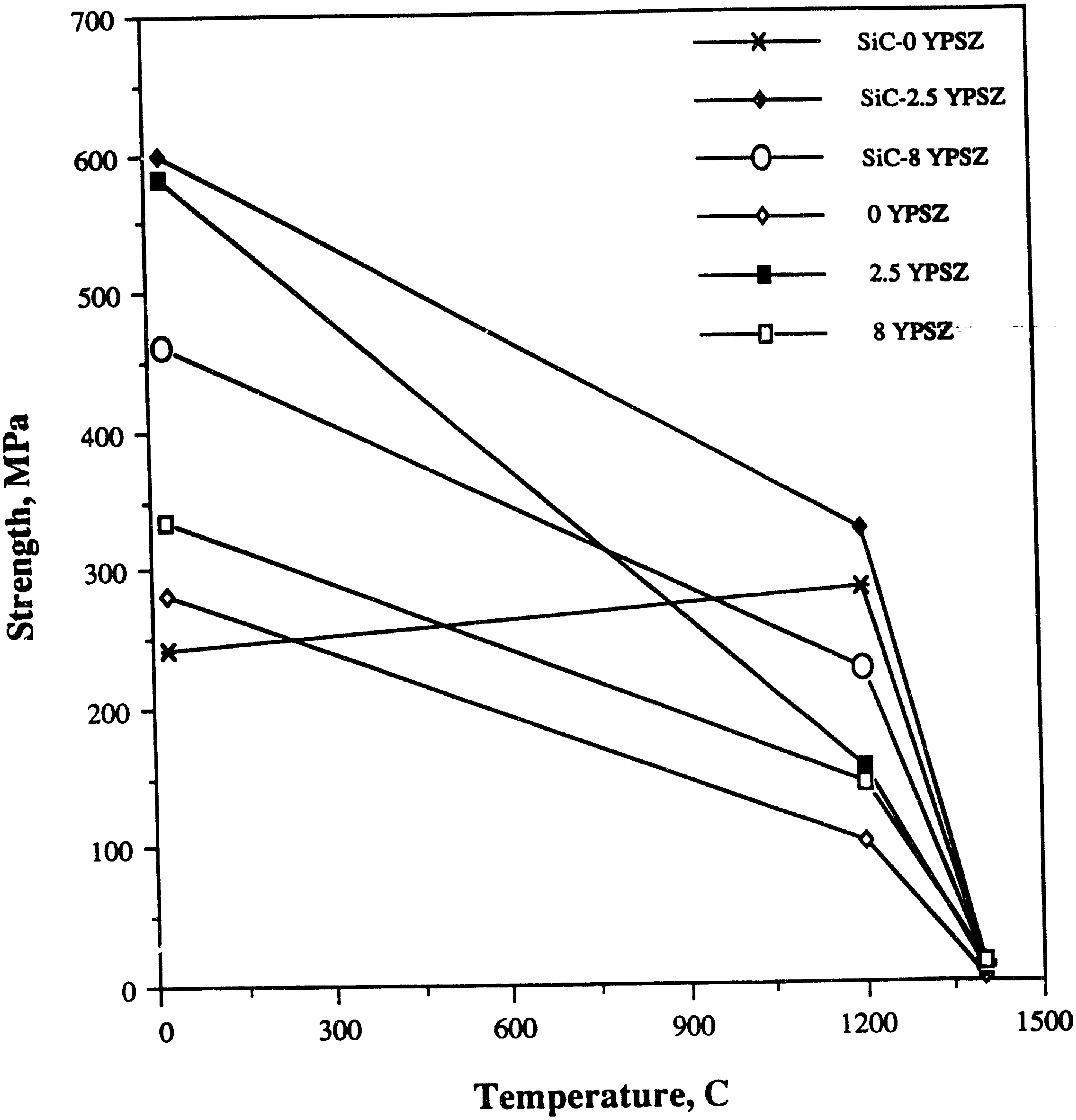


Figure 7: Bend strength versus temperature, for ZrO_2-$MoSi_2$ and ZrO_2/SiC-$MoSi_2$ composites.

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