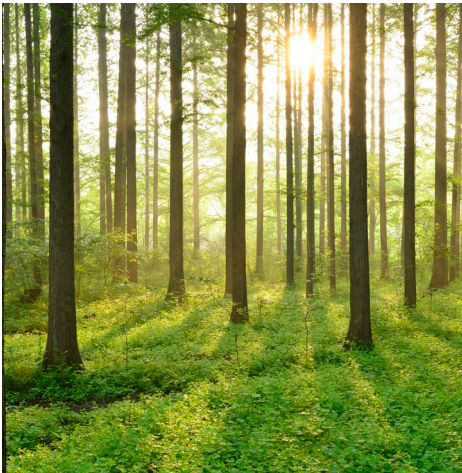


MoSi₂ MATRIX COMPOSITES FOR COMBUSTION COMPONENTS

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MoSi₂ Matrix Composites for Combustion Components

– exposed to high temperature oxidation and hot corrosion

KME 705

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Foreword

The project has been performed within the framework of the materials technology research programme KME, Consortium materials technology for thermal energy processes, period 2014-2018. The consortium is at the forefront of developing material technology to create maximum efficiency for energy conversion of renewable fuels and waste. KME has its sights firmly set on continuing to raise the efficiency of long-term sustainable energy as well as ensuring international industrial competitiveness.

KME was established 1997 and is a multi-cliental group of companies over the entire value chain, including stakeholders from the material producers, manufacturers of systems and components for energy conversion and energy industry (utilities), that are interested in materials technology research. In the current programme stage, eight industrial companies and 14 energy companies participate in the consortium. The consortium is managed by Energiforsk.

The programme shall contribute to increasing knowledge within materials technology and process technology development to forward the development of thermal energy processes for efficient utilisation of renewable fuels and waste in power and heat production. The KME goals are to bring about cost-effective materials solutions for increased availability and power production, improved fuel flexibility and improved operating flexibility, with low environmental impact.

KME's activities are characterised by long term industry and demand driven research and constitutes an important part of the effort to promote the development of new energy technology with the aim to create value and an economic, environmentally friendly and long term sustainable energy society.

Yiming Yao, Chalmers, has been the project leader. Erik Ström and Qi Lu, Kanthal/Sandvik Heating Technology AB, and Johan Ahlström, Chalmers, have been project members. Xin-Hai Li, Siemens Industrial Turbomachinery AB, worked as reference group. Kanthal/Sandvik Heating Technology AB has participated in the project through own investment (60 %) and the Swedish Energy Agency has financed the academic partners (40 %).

The report is a short version of the complete KME report.

Energiforsk would like to thank all the participants for a well performed project.

Bertil Wahlund, Energiforsk

These are the results and conclusions of a project, which is part of a research programme run by Energiforsk. The author/authors are responsible for the content.

Sammanfattning

MoSi₂ ingår som huvudsaklig ingrediens i värmeelement som klarar upp till 1800°C i luft tack vare hög smältpunkt i kombination med mycket bra oxidationsmotstånd. Genom att använda MoSi₂-baserade komposit till komponenter som utsätts för högtemperaturkorrosion skulle potentiellt kunna höja arbetstemperaturen hos en gasturbin med 100-200°C och därmed höja dess verkningsgrad avsevärt. Komponentkostnader kan hållas nere genom pulvermetallurgi följt av trycklös sintring.

Keramiska partiklar såsom ZrO₂ och SiC ökar både brottseghet och krypmotstånd hos MoSi₂. En nackdel med ZrO₂ är att denna blir jonledande vid hög temperatur vilket leder till ökad oxidationshastighet samt att det bildas Mo₅Si₃ i ytan vid sintring vilken försämrar motståndet mot oxidation och termisk cykling. Tillsats av SiC försvårar sintringen pga. låg diffusivitet i denna fas. KME-705 har som mål att förbättra oxidationsegenskaperna hos MoSi₂-ZrO₂-komposit samt att öka sintraddensitet för SiC-förstärkt MoSi₂.

Oxidations- och mekaniska testresultat från KME-705 visade att oxidationsresistensen hos slutsintrad (FS) MoSi₂+15 vol.% ZrO₂-komposit var jämförbar med KS1700-material, uppvisat i termisk cyklisk utmattning (TCF) vid 1200-1300°C och vid isotherm exponering vid 1400°C; krypmotstånd var som för KS HT vid 1600°C. Böjhallfastheten hos FS-kompositen uppnådde 427 MPa vid 1200°C, vilken var 28 % mer än den för enbart gassintrade (AS) motsvarigheten, och brottseghet vid rumstemperatur var 1,5 gånger den för monolitisk silicid. Den högsta sintringstätheten på 97 % av teoretisk uppnåddes för MoSi₂+10 vol.% SiC-komposit. Oxidationsbeteendet hos AS MoSi₂-SiC-komposit vid 1400°C var jämförbart med det för KS1700 och MoSi₂-ZrO₂-komposit; krypmotstånd vid 1600 och 1700°C var jämförbart med Kanthal HT-material och överlägsen MoSi₂-ZrO₂-komposit. Det påvisade också att SiC-tillsatser i kompositmaterialet började brytas ner under exponering vid 1700°C. PLS Mo(Si,Al)₂-SiC-material oxiderades kraftigt och uppträdde sprött i samtliga oxidationsexponeringar i luft på grund av bildandet av tjocka, icke-skyddande (Al,Si)- och Al-oxidskikt.

Sammanfattningsvis kan PLS-FS MoSi₂+15 vol.% ZrO₂ användas för lastbärande komponenter vid 1200°C i oxidationsmiljöer och som skyddskomponenter upp till 1600°C. PLS-AS MoSi₂+10 vol.% SiC-komposit kan användas för luftoxidation och potentiellt för korrosionskomponenter i förbränningsgas (CO + H₂) upp till 1600°C. Mo(Si,Al)₂-SiC-komposit kan ha potential i reducerande och torra förbränningsatmosfärer med höga gashastigheter tack vare att de bildar aluminiumoxid, vilken skyddar bättre än kiseloxid under dessa omständigheter.

Under KME-705 utvecklades Single Edge V-Notch Beam-teknik (SEVNB) för provning av cylindriska tvärsnitt. SEVNB är en standardmetod för seghetsmätning av spröda material. Utmaningarna innefattade att bilda en förspricka med rotradie <20 µm i en cylindrisk provstav och höga krav på linjering av fixturen. Därför tillverkades en noggrann poleringsmaskin för att tillverka anvisningen samt speciella fixturer. Den innovativa tekniken kan appliceras direkt på de tillverkade

cylinderproverna. Hög noggrannhet av K_{IC} -resultat erhöles för kompositmaterialet jämfört med referenser från National Physics Laboratory (NPL) UK. Testmetoden gav pålitliga K_{IC} -mätningar för keramiska material.

Vi kommer att fokusera på tillverkningstekniker av ovanstående kompositmaterial, inklusive beläggningar och additiv tillverkningsteknik.

Summary

MoSi₂ based material is the soundest heating element material owing to its high melting point, excellent oxidation resistance, high thermal conductivity and thermal stability. Applying MoSi₂ matrix composites for hot corrosion components can potentially increase the efficiency of a gas turbine by increasing operation temperature by 100–200°C. In addition, the cost of component manufacturing would be reduced by using standard powder metallurgy (PM) – PressureLess Sintering (PLS) process.

Oxide and non-oxide particles e.g. ZrO₂ and SiC particle additives are efficient as reinforcement to increase toughness and creep resistance of silicides. The challenge is that the ZrO₂ becomes ion-conductive at elevated temperatures and a Mo₅Si₃ surface layer usually forms on MoSi₂-ZrO₂ in PLS, which degrades oxidation and Thermal Cyclic Fatigue (TCF) properties. On the other hand, SiC greatly restricts sintering ability in PLS due to slow diffusion rates. KME-705 was aiming at improving oxidation property of MoSi₂-ZrO₂ composite using final sintering (FS) in air treatment; and improving sintered density of SiC reinforced MoSi₂ and Mo(Si,Al)₂ matrices using PLS process. The improvements were evaluated with high temperature tests.

The oxidation and mechanical testing results from KME-705 showed that oxidation resistance of FS MoSi₂+15 vol.% ZrO₂ composite was comparable to KS1700 material, revealed in TCF tests at 1200 – 1300°C and isothermal exposure at 1400°C; creep resistance was close to state of the art heating element material KS HT in sag test at 1600°C. The flexure strength of the FS composite was 427 MPa at 1200°C, which was 28% greater than that of the as-sintered (AS) counterpart, and the room temperature fracture toughness was 1.5 times that of the monolithic silicide. The highest sintered density, 97% of theoretical, was achieved for MoSi₂+10 vol.% SiC composite. The oxidation behaviour of AS MoSi₂-SiC composite at 1400°C was comparable to that of KS1700 and FS/base MoSi₂-ZrO₂ composite; creep resistance at 1600 and 1700°C was comparable to KS HT material and superior to MoSi₂-ZrO₂ composite. It was also revealed that SiC additives in the composite started to decompose in the exposure at 1700°C. PLSed Mo(Si,Al)₂+SiC materials were severely oxidized and behaved brittle in all the oxidation exposures in air due to the formation and spallation of thick, unprotective (Al,Si)- and Al-oxide scales.

In conclusion, PLS-FS MoSi₂+15 vol.% ZrO₂ can be used for structural components at 1200°C in oxidation environments, and protective components up to 1600°C. PLS-AS MoSi₂+10 vol.% SiC composites can be used for air oxidation and potentially for combustion gas CO+H₂ corrosion components ≤ 1600°C. Alumina former Mo(Si,Al)₂-SiC composites could be promising in reducing and dry combustion gas atmospheres under high gas velocities.

During KME-705, Single Edge V- Notch Beam (SEVNB) technique of cylindrical testing bars was developed. SEVNB is a standard toughness measurement method for brittle materials. The challenges of the innovation included pre-cracking

technique of a V-notch with root radius $<20\ \mu\text{m}$ in cylindrical shaped beams, and a high demand for the testing fixture alignment of the notched beam. Therefore, a precise notch polishing machine was built and special fixtures were made. The technique can be directly applied to as-manufactured cylinder specimens. High accuracy of K_{Ic} results were obtained for the silicide composite materials compared with the references of National Physics laboratory (NPL) UK. The innovation provided reliable K_{Ic} measurements for ceramics materials.

We will focus on investigations on manufacturing techniques of above composite materials, including coatings and additive manufacturing techniques

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1 Background

In high power-to-heat ratio biomass power generation systems, gas turbines are integrated with biomass gasification combined cycle systems (IGCC). The biomass gasification process produces a mixture of CO+H₂ and other gas product (synthesis gas, syngas, or fuel gas) at a high temperature around 800-1000°C, and then, converted into liquid gas fed into a gas turbine combustor. Therefore, a large amount of hazardous species such as H₂S, K, HCl still remains in the fuel gas and leads to an increased risk of corrosion attack on the high temperature components. In currently existing systems, such as in Värnamo, the fuel gas is cooled down to below the dew-point of alkali compounds to minimize degradation. However, this means that there are heat losses in the currently used systems. Thus, the system efficiency can be increased if the system would use as-produced gas. This is a challenge to the components with even severe corrosion in biomass gasification systems in future.

MoSi₂ matrix composites are potential candidates for HT structural and component material for hot corrosion environments, primarily due to their high melting point (2020°C) and service temperature (>1600°C), excellent oxidation resistance, high thermal conductivity, and higher ductility than conventional structural ceramics [1]. However, low fracture toughness (2-3 MPa·m^{1/2}), and reduced strength and creep resistance above 1200°C have to be improved by reinforcements. Zirconia particle additive has efficient toughening effect owing to a phase transformation toughening effect. So far, the greatest improvement in fracture toughness is to 8 MPa·m^{1/2} reported for MoSi₂-ZrO₂-SiC composites [2]. However, hot corrosion resistance in combustion atmosphere has to be validated for this composite.

Regarding non-oxide additives, SiC and Si₃N₄ are commonly used for reinforcement of MoSi₂ composites. The MoSi₂-SiC composites have high maximum service temperature, as the oxide products of MoSi₂ and SiC are not influenced by any eutectics. Intermetallic base MoSi₂-SiC composite, an excellent high temperature oxidation-resistant material meant for aerospace structural applications between 1600 and 1700°C under oxidizing environment, has been developed successfully using powder metallurgy techniques using hot pressing to obtain 98.5% of theoretical density [3]. US aerospace manufacturer Pratt & Whitney has been developing advanced MoSi₂-SiC and MoSi₂-Si₃N₄ for blade outer air seal (BOAS) hot section components used as stationary parts [4]. Gas burner testing has shown that these composites possess significant thermal shock resistance in a simulated jet fuel combustion environment from room temperature to 1500°C. Recently, TCF and petting resistance behaviour of MoSi₂-Si₃N₄ at 500°C, and improved toughness by a factor of 3 in MoSi₂-SiC and MoSi₂-SiC-Si₃N₄ have also been reported [6, 7]. Generally, the composites are generally produced using hot pressing due to poor sinterability of SiC and Si₃N₄. Suitable sintering aids are usually needed, which will risk mechanical property at high temperature. KS ER is an alumina forming Mo(Si,Al)₂ based material that is used as a unique heating element material for dry and aggressive gaseous environments. It is also recommended for carburizing atmosphere, endogas and N₂+H₂ as well as inert

gases at high temperatures [8]. However, its toughness and HT strength can be improved by reinforcement of non-oxide additives.

KME 105-705 projects have been continuously engaged in the development of MoSi₂ matrix composites for hot corrosion applications since 1997. During the collaboration between two industrial companies (Sandvik Heating Technology AB and Siemens Industrial Turbomachinery AB), a series of research has been carried out aiming at developing composite materials and component manufacturing processes. Fracture toughness to 7-8 MPa√m was obtained in MoSi₂-ZrO₂-MoB-SiC composite, with other properties remaining unchanged or improved compared to KS1800 at 1100°C. A practical manufacturing process consisting of CIP – pre-sintering – machining – sintering was developed and a high density heat shield prototype consisting of (Mo,Cr)Si₂-ZrO₂ was made during KME-405. The intrinsic oxidation resistance and TCF property of MoSi₂+15 vol.% ZrO₂ composite was proved being similar to KS materials at 1200 to 1400°C. A detrimental effect of Si depleted Mo-Zr-Si silicide layer on oxidation and TCF properties was found in the as-sintered composite surface. The result in KME-505 showed that the as-sintered surface could be removed and a thin protective glassy layer be formed by a FS pre-oxidation treatment. KME-705 project is continuation and development of KME-505. The TCF test and high temperature mechanical test on the FS MoSi₂-ZrO₂ composite has been completed. For the sake of corrosion resistance for combustion gases, new composites of SiC reinforced MoSi₂ and Mo(Si,Al)₂ matrix composites by PLS has been developed and preliminary exposure testing has been performed.

2 Project group

Yiming Yao, Ph.D, project manager, senior researching engineer, Industrial and Materials Science, Chalmers University of Technology, working with characterization and mechanical testing (6%).

Erik Ström, Ph.D, Principal Engineer, High Temperature Ceramic Materials Research, Sandvik Heating Technology AB, working with material design, processing, high temperature oxidation testing (5%),

Qi Lu, Ph.D, Senior Engineer, High Temperature Ceramic Materials Research, Sandvik Heating Technology AB, working with high temperature oxidation testing (5%).

Johan Ahlström, Docent, Industrial and Materials Science, Chalmers University of Technology, working with mechanical testing (1%).

Xin-Hai Li, Ph.D, Siemens Industrial Turbomachinery AB, Principal Engineer, working as reference group (no charged cost).

3 Results

During KME-705, we have fulfilled following work:

1. MoSi₂+15 vol.% ZrO₂ composite was produced by extrusion followed by Pressure Less Sintering (PLS) and was heat-treated with final sintering (FS) in air. Thermal Cyclic Fatigue (TCF), high temperature mechanical properties and creep resistance evaluation (sag test) were conducted on this composite.
 - Composite with high sintered density (98% of theoretical density, T.D.) and uniform particle dispersion was produced with the process. Final sintering removed the as-sintered Mo₅Si₃ surface layer, having poor oxidation resistance, which was replaced with a thin protective silicon oxide layer that formed on the composite surface.
 - The final sintered composite exhibited excellent protective oxidation behaviour in TCF tests at 1200°C-1300°C and isothermal oxidation exposure at 1400°C, comparable to Kanthal Super 1700 material.
 - Sag test revealed that the FS composite retained its shape and oxidation resistance at 1600°C, and was deformed at 1725°C to similar extent as for state-of-the-art Kanthal Super HT.
 - The room temperature flexure toughness K_{IC} of the FS composite was 4.36 MPa·m^{1/2}, which was 1.5 times of that of monolithic silicide. The flexure strength of the FS composite was 427 MPa at 1200°C, which was 28% greater than that of the as-sintered (AS) counterpart. In addition, the composite exhibited 2.6% strain without fracture at this temperature.
2. SiC particles reinforced composites MoSi₂-SiC and Mo(Si,Al)₂+SiC were produced with SiC additive contents of 5, 10, 15 vol.%. Isothermal oxidation exposure and sag test were performed:
 - SiC plays a positive role in protecting the sintered surface from forming Mo₅Si₃ layer in H₂ and Ar sintering atmospheres in both MoSi₂-SiC and Mo(Si,Al)₂-SiC. The fracture toughness K_c was not affected by SiC additives, but hardness HV was low due to the low sintered density.
 - Thin protective SiO₂ scales were formed directly on AS MoSi₂-SiC materials at 1400°C. Long-term exposure at 1400°C resulted in further sintering that increased HV and K_c. The oxidation behaviour at 1400°C of MoSi₂-SiC materials was comparable with that of KS1700 and MoSi₂-ZrO₂ composite.
 - Sag test at 1700°C in air showed that creep resistance of MoSi₂-SiC (MoSi₂+10 vol.% SiC) was comparable to KS HT material, which is

superior to MoSi₂-ZrO₂ composite. However, it was also found that SiC additives were decomposed at 1700°C, which created glass bubbles and pores on the surface due to the released gases from the decomposition.

- PLSed Mo(Si,Al)₂+SiC materials were severely oxidized and behaved brittle in all the oxidation exposures in air due to the formation and spallation of non-protective and thick (Al,Si)- and Al-oxide scales.

In brief:

(1) FS process improves high temperature oxidation resistance and mechanical properties of PLS produced MoSi₂-ZrO₂ composite. FS MoSi₂+15 vol.% ZrO₂ can be used for structural components over 1200°C in oxidation environments, and oxidation protection components up to 1600°C.

(2) AS MoSi₂-SiC composites (SiC additive < 10 vol.%) with high oxidation resistance and creep resistance can be used for air oxidation and perhaps CO+H₂ corrosion components up to 1600°C.

(3) Mo(Si,Al)₂-based composites are not suitable for high temperature applications in air environments. These composites could be more promising for applications in reducing atmospheres, under high gas velocities (as Al₂O₃ is not volatile such as SiO₂), and potentially in dry combustion gases.

With the results of KME 705, two groups of PLS produced silicide composites (oxide and carbide additives) i.e. MoSi₂+15 ZrO₂ (FS) and MoSi₂+10 SiC (AS) can be recommended for high temperature corrosion applications, which can allow to increase the efficiency of the engines in terms of increasing the operation temperature by 100-200°C and reducing losses due to cooling that is employed today. These composites can potentially substitute for Ni-based superalloys, allowing for critical metals, e.g. Ni, to be saved, which is greatly appreciated for resource protection and sustainable development. Furthermore, using powder metallurgy and PLS for silicide composites is relatively simple and economical compared with expensive Ni-based alloys regarding the raw material price and complicated manufacturing processes.

3. Single Edge V- Notch Beam (SEVNB) technique was developed to allow for testing of cylindrical samples during KME-705. SEVNB is a standard method for toughness testing of brittle materials, in which 3-point or 4-point bending testing is performed on a testing beam with a pre-cracked sharp V-notch. The ASTM standard for SEVNB usually requires a rectangular cross section of the sample, which is difficult and requires extra machining for the hard and brittle materials. The challenges of the innovation included pre-cracking technique of a V-notch with root radius <20 μm on a cylindrical sample, and a high demand for the testing fixture alignment of the notched beam. Therefore, a precise notch polishing machine, and special fixtures for 3-point and 4-point bending tests were

made for the project. This technique can be directly applied to as-manufactured cylindrical specimens, and does not require further machining of the samples. High accuracy of K_{Ic} results (accuracy 0.7%) was obtained compared with reference tests performed at National Physics Laboratory (NPL), UK. A considerable deviation was believed to attribute from the too high loading scale of the loading cell for metallic materials currently used in the lab. The innovation is not only a breakthrough for the K_{Ic} measurement technique for the project, but is also valuable and beneficial for the industry that produces as well as develops ceramics and intermetallic materials.

4 Discussion

According to the project plan, the TCF/isothermal exposure in H₂-containing atmosphere at 1200°C, and toughness K_{1C} testing were not conducted for the Mo(Si,Al)Si₂+SiC composites. One of the reasons was poor oxidation behavior of the composites in air, which might result from too high Al addition to the base MoSi₂ silicide. Another reason was the unavailability of high temperature exposure device for H₂-atmosphere. As a substitution, the MoSi₂-SiC composites were, therefore, produced and tested at high temperatures in air, and the result was described in this report.

Investigations from other groups in KME community have found that alumina forming materials such as Kanthal APM and aluminide coatings have excellent corrosion resistance in lab and field exposure tests in biomass and synthesis gas (H₂, CO, CO₂, CH₄, N₂) at 450-1100°C, contrasted to standard stainless steels and Ni-based materials that must be protected. Mo(Si,Al)₂ composite is one of the potential choices to compete with APM and aluminides under synthesis gaseous environment.

It has been revealed in KME-705 that carbon solubility in the silicide of MoSi₂-SiC reached 2.6 wt.% when sintered in H₂ at 1600°C, which allowed the composites to exhibit both high reducing-gas and high oxidation resistance at high temperature. Therefore, the MoSi₂-SiC and Mo(Si,Al)₂-SiC composites and composite coatings on medium- and high carbon steels could potentially be an alternative, and obviously there is a fundamental and technical interest in the corrosion of C/Al-containing MoSi₂ composite coatings. The corrosion resistance of the composites in H₂, CO, CO₂, CH₄, N₂ gases needs to be investigated. Meanwhile, coating processes such as plasma spray or laser cladding [9] should be developed and compared with the properties of aluminide coatings.

Regarding the high temperature component material and manufacturing, MoSi₂-ZrO₂ with improved mechanical and oxidation properties is promising. But, the toughness is still a node of manufacturing and engineering even though it is improved by ZrO₂ reinforcement. Additive Manufacturing (AM) could be a solution. Successful net-shape manufacturing of brittle materials and ceramics using AM, e.g. Selective Laser Melting (SLM) and Selective Laser Sintering (SLS) has been drawn wider attentions in ceramic community in recent years [10].

Currently, we have no further plans in KME projects. But we will continue the investigations through other funds and collaborations. The AM and graded functional materials for high temperature corrosion could be in consideration.

5 Publications

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Appendix

Energimyndighetens titel på projektet – svenska	
MoSi ₂ -matriskompositer för förbränningskomponenter utsatta för högttemperaturkorrosion	
Energimyndighetens titel på projektet – engelska	
MoSi ₂ matrix composites for combustion components exposed to high temperature oxidation and hot corrosion	
Universitet/högskola/företag	Avdelning/institution
Chalmers University of Technology	Industrial and Materials Science
Adress	
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Namn på ev övriga projektdeltagare	
Erik Ström	
Nyckelord: 5-7 st	
Silicide, composites, high temperature materials, corrosion, mechanical property	

MoSi₂ MATRIX COMPOSITES FOR COMBUSTION COMPONENTS

MoSi₂ based material is the soundest heating element material owing to its high melting point, excellent oxidation resistance, high thermal conductivity and thermal stability. Applying MoSi₂ matrix composites for hot corrosion components can potentially increase the efficiency of a gas turbine by increasing operation temperature by 100–200°C.

The oxidation resistance of MoSi₂+15vol%ZrO₂ composite after final sintering was comparable to KS1700 material in cyclic exposure at 1200 – 1300°C and isothermal exposure at 1400°C; the creeping resistance was close to the state of KS HT material in sag testing at 1600°C; the flexure strength retained 427 MPa at 1200 C. The oxidation property of as-PLS sintered MoSi₂+10vol%SiC composite at 1400°C was comparable to that of KS1700 and FS MoSi₂-ZrO₂ composite; the creeping resistance at 1600°C was comparable to that of Kanthal HT material and superior to MoSi₂+ZrO₂ composite.

Single Edge V- Notch Beam toughness measurement technique was developed for cylindrical shaped-beam. The technique can be directly applied to as-manufactured testing pieces without machining. High accuracy of KIC data were obtained compared with the references of National Physics laboratory, UK. The innovation provided reliable KIC measurement for R&D of ceramics/ intermetallics materials.

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