

- Applying organization

Microstructure and Surface Analysis, Materials and Manufacturing Technology, Chalmers University of Technology

- Organisation number (Organisationsnummer)

556479-5598

- Project leader

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- Co-applicants

Sandvik Heating Technology AB, Erik Ström

- Project

Continuation of a previous project KME-505

- Title

MoSi₂-matriskompositer för förbränningskomponenter utsatta för hot korrosion

Förslaget syftar till att utveckla avancerade MoSi₂-matriskompositer för varmkorrosionskomponenter för förbränningskammaren i gasturbinmotorer. Målet är att göra kompositmaterial med varmkorrosionsbeständighet och motstånd mot termisk cykling i förbränningsmiljön och måttliga mekaniska egenskaper vid 1200°C. Två typer av kompositer kommer att förberedas i projektet med hjälp av pulvermetallurgi (PM) och trycklös sintring (PLS): (1) föroxiderad MoSi₂-ZrO₂ komposit, och (2) föroxiderad Mo(Si,Al)₂-SiC komposit. Materialen kommer att utvärderas med cyklisk oxidation (TCF) i luft och isotermisk exponering i H₂-innehållande atmosfär vid 1200°C. Mekanisk hållfasthet hos kompositerna kommer att testas vid 1200°C, och jämföras med konventionella keramiska material. Projektet är ett samarbete mellan Sandvik Heating Technology AB (SHT) och Chalmers Tekniska Högskola (CTH).

MoSi₂ matrix composites for combustion components exposed to high temperature oxidation and hot corrosion

The proposal aims at developing advanced MoSi₂ matrix composites for hot corrosion components in a combustion chamber of gas turbine engines. The goal is to make composite materials having resistance to hot corrosion and thermal cycling in the combustion environment and moderate mechanical properties at 1200°C. Two types of composites will be prepared in the project using powder metallurgy (PM) and pressure-less sintering (PLS) techniques: (1) pre-oxidation treated MoSi₂-ZrO₂ composite, and (2) pre-oxidized Mo(Si,Al)₂-SiC composite. The materials will be investigated with cyclic oxidation (TCF) testing in air and isothermal exposure in H₂-containing atmosphere at 1200°C. The flexure strength will be tested at 1200°C, and will be compared with that of conventional ceramic materials. The project is collaboration between Sandvik Heating Technology AB (SHT) and Chalmers University of Technology (CTH).

- Motivation

The purpose of Materials technology for thermal energy processes program (KME) is to use materials technology development to promote efficient use of renewable fuels and waste fractions in the production of power and heat in thermal processes involving high fuel flexibility, good part-load characteristics and minimal impact on the environment. Research is essential for developing materials that are sustainable and resistant to erosion and corrosion in different environments. The KME goals include evaluations of exposures and

application tests of various conventional solid and composite materials and/or coated materials for the purpose of greater fuel flexibility and increased electricity production, and to test and validate new materials for future industrial gas turbines that permit higher fuel flexibility, availability and efficiency, as well as cyclic operation.

The gaseous fuels with wider availability and lower price contain high concentrations of solids, water, higher hydrocarbons, hydrogen sulphide, CO_2 and CO , and hydrogen, etc., resulting in enhanced corrosion on the high temperature components of a gas turbine. The maximum operating temperature of currently used conventional stainless steels and Ni-superalloys is hardly over 1050°C , limited by their melting points. MoSi_2 based material is the soundest heating element material owing to its high melting point, excellent oxidation resistance, high thermal conductivity and thermal stability. The proposed project will investigate MoSi_2 matrix composites reinforced with oxides and non-oxide ceramics as substitute for conventional materials in suitable sections of a gas turbine, aiming at prolonging component lifetime in hot corrosion environments.

Energy relevance: the proposed MoSi_2 matrix composites for hot corrosion components allow the components being operating above 1200°C without cooling. In particular, $\text{MoSi}_2\text{-ZrO}_2$ has fairly good TCF mechanical property at 1200°C , and Kanthal Super materials are the most durable materials in oxidising and H_2 -containing environments with operation temperatures up to 1100°C , or even higher without cooling, contrasted to other hot corrosion metallic and refractory materials. Thus, it is believed that the energy consumption will be considerably reduced and the electricity production efficiency will be increased by applying MoSi_2 composite components at suitable parts in the gas turbine. As MoSi_2 based materials produced by PM-PLS production are cheaper than conventional Ni-based superalloys, a potential up-scale of the materials in applications can be expected.

Industry relevance: Sandvik Heating Technology AB (formerly Kanthal) is a worldwide MoSi_2 heating element producing company. The investigation of the materials and methods will potentially provide the company with new MoSi_2 composite products having considerably improved TCF resistance and mechanical properties. For example, development of techniques such as pre-oxidation treatment, modified toughness measurement method, and composite manufacturing for producing components having complicated geometry, will be beneficial and valuable for the industry in manufacturing and developing other ceramics and intermetallics using the PLS technique.

General application: The investigation of $\text{MoSi}_2\text{-ZrO}_2$ and $\text{Mo}(\text{Si,Al})_2\text{+SiC+Si}_3\text{N}_4$ materials will provide data from high temperature corrosion in combustion environments being relevant to the MoSi_2 material family. The results can be used for developing intermetallic coatings for combustor components in new projects, and the knowledge will be valuable to other KME groups that are working with ceramic coatings.

The project will be closely collaborated between Sandvik Heating Technology AB (SHT) and Chalmers University of Technology (CTH). The former is responsible for material processing and HT oxidation testing and the latter is responsible for mechanical testing and characterization. The investigation on MoSi_2 based materials has been continuously conducted during 5 periods of KME projects since 90's. The project members include senior researchers from university and industry, who have profound experience and competence on the studied material. The extensive research work in the proposed project will create 1 to 2 master programs for University students.

• 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+H_2 and other gas product (synthesis gas, syngas, or fuel gas) at a high temperature around $800\text{-}1000^\circ\text{C}$. The product gas is then converted into a relatively cleaned liquid gas that is fed into a conventional gas turbine combustor where it is fired with air to generate combustion products with high temperature and pressure. Therefore, a large amount of hazardous compounds such as H_2S , K, HCl still remains in the gas and leads to an increased risk of corrosion attack on the component materials. 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 would result in higher operating temperature but also challenge the components with even more severe corrosion in future biomass gasification systems.

MoSi_2 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 ($>1600^\circ\text{C}$), excellent oxidation resistance, high thermal conductivity, and higher ductility than conventional structural

ceramics [¹, ²]. 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 [³]. 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 reported for MoSi₂-ZrO₂-SiC composites [⁴, ⁵]. However, hot corrosion resistance in combustion atmosphere has to be validated for this composite.

Regarding non-oxide reinforced MoSi₂ composites, SiC and Si₃N₄ are the most famous high temperature ceramics having high temperature corrosion resistance for structural applications. The MoSi₂-SiC composites have maximum service temperature of 1800°C, since SiO₂ is the oxidation product of both MoSi₂ and SiC and the system is 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 [⁶]. 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, which are located directly opposite to the rotating hot section turbine blades for its gas turbine engines [⁷]. Gas burner testing has shown that MoSi₂-SiC and MoSi₂-Si₃N₄ composites possess significant thermal shock resistance in a simulated jet fuel combustion environment from room temperature to 1500°C. Recent work at NASA Lewis Research Center (Cleveland, OH) has concentrated on MoSi₂-Si₃N₄ composites reinforced with SiC continuous fibers that present with significant absorption of impact energy at both room temperature and elevated temperatures [⁸]. Recently, TCF and peeling resistance behavior at 500°C in MoSi₂-Si₃N₄ [⁹], and improved toughness by a factor of 3 in MoSi₂-SiC and MoSi₂-SiC-Si₃N₄ have also been reported [¹⁰]. Information on high temperature corrosion behavior in combustion chamber environment of industrial turbines is currently not available for this material family and has, thus, to be tested. In addition, the composites are generally produced using hot pressing due to poor sinterability of SiC and Si₃N₄. Suitable sintering aids are needed, which will risk the strength and creep resistance at high temperature.

Kanthal Super ER is an alumina forming Mo(Si,Al)₂ based material that is used as a unique heating element material for aggressive gaseous environments [¹¹]. The operating temperature of Kanthal Super ER in dry H₂ can be as high as 1550°C at a dew point (D_p) of -40°C. It is also recommended for carburizing atmosphere, endogas and N₂+H₂ as well as inert gases at high temperatures. This alumina scale former has been extensively investigated in other HTC projects. The excellent corrosion resistance of the material at low and high temperature is proved. However, its toughness and HT strength has to be improved by reinforcement, e.g. SiC. Again, the applicability of PLS method for SiC containing composite is the challenge.

KME programs have been continuously engaged in the development of MoSi₂ matrix composites for hot corrosion applications since 1997. With collaboration of two industrial companies (Sandvik Heating Technology AB and Siemens Industrial Turbomachinery AB), a series of researches have been carried out aiming at developing MoSi₂-based composites and component manufacturing process for gas turbine prototypes. An improvement in fracture toughness to 7-8 MPa·m has been achieved in MoSi₂-ZrO₂-MoB-SiC composite, with other properties remaining unchanged or improved e.g. Hv, flexure strength and creep rate compared with Kanthal Super 1800 at 1100°C. A practical manufacturing process consisting of CIP – pre-sintering – machining – final sintering has been developed during KME-405. A high density heat shield prototype with a complicated shape has been made using Cr alloyed (Mo,Cr)Si₂-ZrO₂ (Figure 1). Mechanical testing at 1200°C has showed flexure strength of this composite similar to that of Si₃N₄, and better ductility than Si₃N₄ at this temperature. The intrinsic oxidation resistance properties of MoSi₂+15 vol.% ZrO₂ composite similar to Kanthal Super materials have been proven during isothermal exposure at 1400°C for 1000 h and TCF at 1200 and 1300°C. A detrimental effect of Si depleted Mo-Zr-Si silicide layer on oxidation and TCF properties has been found in the as-sintered surface resulting from reduction of the zirconia oxide additives. It is not practical to remove this hard layer using mechanical grinding for future component applications. A preliminary result has showed that a thin protective glassy layer can be formed on the composite surface after pre-oxidation treatment.

The proposed project is a continuation of former KME-505 project. According to the previous results, zirconia reinforced MoSi₂ composite with excellent TCF resistance and moderate mechanical properties could be suitable for components in the combustion chamber, particularly in the pilot burner where high concentrations of fuel gas are mixed with oxygen prior to combustion, and severe corrosion often occurs in short service time for the component [¹²]. The TCF and HT mechanical test on the pre-oxidised material will be completed for this composite. For the sake of corrosion resistance, new composite of Al alloyed MoSi₂ matrix reinforced with SiC produced by PLS and in-situ sintering method is proposed. The material manufacturing facility, PLS process know-how and oxidation testing are available in SHT. The project will be implemented in collaboration between SHT and CTH; the latter will perform mechanical testing and characterization.

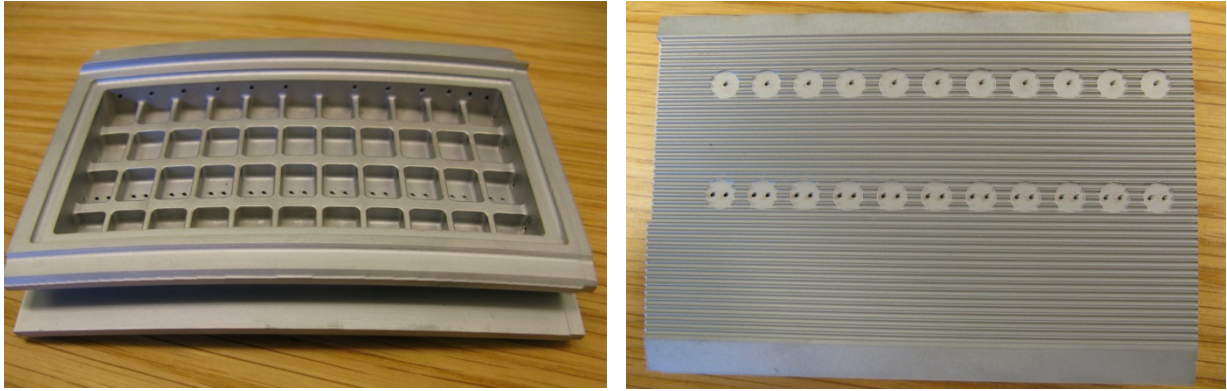


Figure 1. A full-size heat shield prototype made with $(\text{Mo}_{0.9}\text{Si}_{0.1})\text{Si}_2 + 15 \text{ vol.}\% \text{ ZrO}_2$ for a gas turbine (dimensions $220 \times 61 \times 15 \text{ mm}$), with sintered density 96% of T.D., developed during KME-405. The left is the front side, right is the back side.

• Goal

The proposed project will be continuation and extension of the work in KME-505. The goal is to produce high corrosion resistance MoSi_2 based composite for gas-turbine combustion chamber components at operation temperature of 1200°C .

Al -alloyed MoSi_2 will be reinforced with SiC particles using PLS and in-situ reaction method.

High corrosion resistance in oxidation and H_2 atmospheres at 1200°C , improved room temperature toughness by a factor of 2 - 3, and flexure strength of 300 MPa at 1200°C is expected to be achieved for the proposed materials.

Following investigations will be implemented:

- (1) Finish the investigation on the effect of pre-oxidation on TCF and HT mechanical properties of $\text{MoSi}_2 + 15 \text{ vol.}\% \text{ ZrO}_2$ composite produced by CIP – PLS process. TCF and flexure testing will be performed at 1200°C
- (2) Prepare $\text{Mo}(\text{Si},\text{Al})_2$ matrix composites reinforced with SiC with PLS and pre-oxidation treatment.
- (3) TCF in air and isothermal exposure in H_2 -containing atmosphere will be performed at 1200°C .
- (4) Flexure and toughness K_{IC} testing will be conducted at room temperature and 1200°C .

• Project plan

1. Pre-oxidation and testing of $\text{MoSi}_2 - \text{ZrO}_2$ composite

Pre-oxidation treatment will be performed for PLS produced $\text{MoSi}_2 + 15 \text{ vol.}\% \text{ ZrO}_2$ composite (MZ) composite. A Kanthal Super technique will be adopted, in which the as-sintered composite is heated up in a short time to $1700\text{--}1750^\circ\text{C}$ in air, and a protective scale is formed rapidly. The pre-oxidation can be performed with resistive heating or furnace heating. The preliminary result is promising (Figure 2). A uniform SiO_2 scale with thickness of 3-4 microns was formed on MZ composite surface by resistive heating. Further pre-oxidation experiments will be carried out in furnace heating conditions. The material process will be conducted at SHT.

The high temperature tests to be performed at 1200°C include: flexure strength and toughness, isothermal exposure in $\text{H}_2 + \text{N}_2$ atmosphere for 1000 h, and TCF in air for 1000 cycles. The pre-oxidation, isothermal exposure and TCF oxidation will be conducted at Sandvik Heating Technology; room temperature mechanical measurements and microstructure characterization will be conducted at Chalmers, and high temperature mechanical testing will be performed in NPL, UK, via Chalmers. The results will be published as conference papers or in international journals.

2. Preparation and testing of $\text{Mo}(\text{Si},\text{Al})_2 - \text{SiC}$ composite

$\text{Mo}(\text{Si},\text{Al})_2 - \text{SiC}$ composite (MAS) with SiC content of 5, 10, 15 vol.% will be prepared with the Kanthal Super ER material as matrix, and Si and C elemental powder or SiC powders. The matrix of ER consists of C40-phase $\text{Mo}(\text{Si},\text{Al})_2$ (with Al content of 30 at%), Al_2O_3 and $\text{Mo}_5(\text{Si},\text{Al})_3$. The $\text{Mo}(\text{Si},\text{Al})_2$ matrix powder will be mixed with Si and C elemental powders or SiC powders according to different SiC volume fraction in a conventional ball mill and a centrifugal planetary ball mill used for mechanical alloying (MA). The powder mixture will be consolidated using CIP followed by PLS sintering in Ar. Pre-oxidation treatment will be performed after PLS

sintering. Silica protective scale will be formed, and the PLS piece will be further densification in suitable treatment temperature and time. The material process will be conducted at SHT.

The final solution for the densification of $\text{Mo}(\text{Si,Al})_2\text{-SiC}$ composition should use hot pressing or PLS sintering with the powder synthesized with MA-SHS (mechanically assisted self-propagating high temperature synthesis) [13].

The same tests will be performed for MAS composite. The high temperature tests to be performed at 1200°C include: flexure strength and toughness, isothermal exposure in H_2+N_2 atmosphere for 1000 h, and TCF in air for 1000 cycles. The pre-oxidation, isothermal exposure and TCF oxidation will be conducted at Sandvik Heating Technology; room temperature mechanical measurements and microstructure characterization will be conducted at Chalmers, and high temperature mechanical testing will be performed in NPL, UK, via Chalmers. The results will be published as conference paper or in international journals.



Fig.2 Pre-oxidized $\text{MoSi}_2+15 \text{ vol.}\% \text{ZrO}_2$ bars for flexure strength and K_{IC} testing. Notice that a glassy surface coating has been formed

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Robert Pompe, Professor Ph.D, Goceram Industrial Ventures AB (industry reference group, no charged costs)

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- Time schedule

Starting date: 2014 09 15

Finishing date 2018 04 15

Final report: 2018 03 31

Finishing account report: 2018 03 31

Status reports: End of each year

Main milestones:

Year	Action
2014 0915 – 2015 0315	Pre-oxidization of MZ using resistive heating and furnace heating. Room temperature flexure and toughness tests of pre-oxidized MZ. Preliminary preparation of MAS powders and characterization.
2015 0315 – 2015 0631	HT flexure strength σ_f and fracture toughness K_{IC} at 1200°C for MZ. Preparation of MAS powders and preliminary sintering tests.
2015 0631 – 2015 1231	Isothermal exposure in air for pre-oxidized MZ and MAS at 1200°C for 1000 h Isothermal exposure in H ₂ -containing atmosphere for MZ and MAS at 1200°C for 1000 h Conference paper for pre-oxidized MZ.
2016 0315 – 2016 0631	Preparation of MAS with different SiC contents. PLS sintering tests.
2016 0631 – 2016 1231	TCF testing of MZ and MAS at 1200°C for 1000 h.
2017 0315 – 2017 1231	Optimization of process and SiC contents for MAS. HT flexure strength σ_f and fracture toughness K_{IC} at 1200C for MAS.
2018 1230 – 2018 0331	Depending on the result, a prototype of burner head might be made with the optimized material. Paper for MAS, and finishing report.

• Industrial reference and financing

Industry reference: Sandvik Heating Technology AB

Industry financing: 150 kkr/year for 4 years (60%)

• Costs

Industry reference: Sandvik Heating Technology AB

Industry financing: 150 kkr/year, total 600 kkr for 4 years (60%)

• Costs

Total cost from KME to the project is 100 kkr / year (40%), 400 kkr for 4 years.

	SUMMA	2014	2015	2016	2017	2018
Lönekostnad	213 480	26600	53 400	53400	53400	26680
Konsultkostnad	24 000	3000	6 000	6000	6000	3000
Utrustning	0	0	0	0	0	0
Material	0	0	0	0	0	0
Laboratoriekostnad	0	0	4000	4000	4000	0
Datorkostnad	0	0	0	0	0	0
Resor	20 000	4400	6600	6600	6600	4400
Övrigt	0	0	0	0	0	0
Indirekta kostnader	121 920	15240	30 480	30480	30480	15240
SUMMA	400 000	49 240	100 480	100 480	100 480	49 320

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Bilaga till projektansökan inom Samverkansprogrammet Materialteknik för term Fördelning av kostnader för varje ingående part

Sökande: Yiming Yao

Projekttitel: KME-705

Part		Kostnadsfördelning									
		Lön	Konsult	Utrustning	Material	Laboratorie	Dator	Resor	Övrigt	Indirekta	Summa
Chalmers University of Technology	2014	26 600	3 000	0	0	0	0	4 400	0	15 240	49 240
	2015	53 400	6 000	0	0	4 000	0	6 600	0	30 480	100 480
	2016	53 400	6 000	0	0	4 000	0	6 600	0	30 480	100 480
	2017	53 400	6 000	0	0	4 000	0	6 600	0	30 480	100 480
	2018	26 680	3 000	0	0	0	0	4 400	0	15 240	49 320
	Summa	213 480	24 000	0	0	12 000	0	28 600	0	121 920	400 000
Sandvik Heating Technology	2014	70 000		2 000	4 000			3 000		24 500	103 500
	2015	100 000		4 000	5 000			5 000		35 000	149 000
	2016	100 000		5 000	5 000			5 000		35 000	150 000
	2017	100 000		5 000	5 000			5 000		35 000	150 000
	2018	30 000		2 000	3 000			2 000		10 500	47 500
	Summa	400 000	0	18 000	22 000	0	0	20 000	0	140 000	600 000
	2014										0
	2015										0
	2016										0
	2017										0
	2018										0
	Summa	0	0	0	0	0	0	0	0	0	0
	2014										0
	2015										0
	2016										0
	2017										0
	2018										0
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	2015										0
	2016										0
	2017										0
	2018										0
	Summa	0	0	0	0	0	0	0	0	0	0
	2014										0
	2015										0
	2016										0
	2017										0
	2018										0
	Summa	0	0	0	0	0	0	0	0	0	0
	2014										0
	2015										0
	2016										0
	2017										0
	2018										0
	Summa	0	0	0	0	0	0	0	0	0	0

STÖD FÖR REGISTRERING I ENERGIMYNDIGHETENS WEBBPORTAL

Följande siffror kan användas för att fylla i projektets ansökan på Energimyndighetens webbportal E-kanalen. Tabellen skapas automatiskt utifrån ovan:

	SUMMA	2014	2015	2016	2017	2018
Lönekostnad	613 480	96600	153 400	153400	153400	56680
Konsultkostnad	24 000	3000	6 000	6000	6000	3000
Utrustning	18 000	2000	4 000	5000	5000	2000
Material	22 000	4000	5 000	5000	5000	3000
Laboratoriekostnad	12 000	0	4 000	4000	4000	0
Datorkostnad	0	0	0	0	0	0
Resor	48 600	7400	11 600	11600	11600	6400
Övrigt	0	0	0	0	0	0
Indirekta kostnader	261 920	39740	65 480	65480	65480	25740
SUMMA	1 000 000	152 740	249 480	250 480	250 480	96 820