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Effect of basalt addition on tribological performance of FeCrSiB HVOF coatings

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Abstract. Products made of basalt are recently gaining sufficient attention due to depletion of raw materials serving as reinforcement for construction, and wear resistant materials. Basalt has sufficient mechanical strength, high hardness, low density and superior corrosion resistance. Basalt and other minerals are used in thick metal coatings to adjust their thermal expansion to that of the steel substrate. High velocity oxygen fuel (HVOF) sprayed coating method allows to prepare solid basalt-steel composite materials since the melting temperatures of basalt and steel are very close and materials obtained by casting or sintering will have low mechanical properties. The aim of the current work is to study the change in tribological response under erosive, abrasive, reciprocal and continuous sliding conditions when basalt is added to the harder matrix. It was found that the addition of 12 vol % of basalt to the FeCrSiB HVOF sprayed coating is generally unfavourable in tribological conditions due to the brittleness of basalt and its flake shape. The worst relative performance was observed in reciprocal sliding conditions and the best one in mild conditions of continuous sliding. Discussion on the mechanisms of materials performance is based on SEM and EDS results.

Key words: basalt, FeCrSiB, HVOF spray, wear, elevated temperature.

1. INTRODUCTION

Products made of basalt (mineral) are recently gaining sufficient attention due to the depletion of raw materials for production of reinforcement for constructional and wear resistant materials (tungsten carbide for cermets, steels for metal-polymer composites and reinforced concrete, etc) [1–5]. Basalt has sufficient strength, high hardness, low density and superior corrosion resistance. It has sufficiently lower price level comparing to tungsten carbide and lower than that of alloys. Basalt and other mineral additions are also used in thick metal coatings to adjust their thermal expansion to that of the steel substrate [6,7]. High velocity oxygen fuel coating method is one of the methods allowing to prepare solid basalt-steel composite materials since the melting temperature of basalt and steels are very close and materials obtained by casting or sintering will have low mechanical properties. HVOF spraying allows to minimize solubility between phases while providing coatings with low porosity suitable for elevated temperatures that are required for high efficiency of thermal processes in energy applications.

Favourable effect of the basalt addition on the wear resistance has been documented mostly for composites when basalt was harder than the binder material (aluminium, plastics, etc) and basalt addition gave rise to the total hardness [1–5]. The aim of the current work is to study the change in tribological response under erosive, abrasive, reciprocal and continuous sliding conditions when basalt is added to the harder matrix. Minerals exhibit brittle behaviour under shock loading conditions. Testing in a wide range of conditions was required to provide information for future research, directed to make materials where mineral additions are favourable for improving the thermal expansion coefficient, corrosion resistance and also the resistance to wear.

2. MATERIALS AND EXPERIMENTAL DETAILS

2.1. Materials

Coatings were applied by the HVOF spray method onto flat C45 (EN 10083, SAE1045) unalloyed carbon steel (0.45 C, 0.60 Mn, 0.30 Si, balance Fe; wt %) substrate of the size 25 × 50 mm. Thickness of the substrate was 10 mm. JP-5000 HVOF *TAFA* system with the 5220 spray gun were used for deposition of coatings. Main parameters of the HVOF spray process are given in Table 1. Mean thickness of the coating was 300–400 μm. FeCrSiB and FeCrSiB-12 vol % basalt coatings were prepared. FeCrSiB self-fluxing alloy powder (13.7 Cr, 2.7 Si, 3.4 B, 2.1 C, 6 Ni, balance Fe; wt %) with particles of spherical shape and size of 10–45 μm was supplied by Höganäs AB. Basalt powder of 25–45 μm size was produced in the Laboratory of Disintegrator Technology of Tallinn University of Technology by disintegrator milling from wastes of different dimensions and shapes, remaining from basalt production routine (Fig. 1). It was found that basalt was melted during thermal spraying and is well incorporated into the steel matrix (Fig. 2). Initial content of basal in the powder mixture was 25 vol %. Actual content of basalt (12 vol %) was verified using SEM image according to ASTM E112-10. Steel droplets are able to remove some of the basalt from the surface during deposition due to their high kinetic energy (steel has density about 3 times higher than basalt) that results in reduced basalt concentration in final coating comparing to initial powder. Composition of basalt (wt %) determined by EDS was found to be as follows: 51.8 O_2, 1.7 Na, 2.2 Mg, 8.2 Al, 24.1 Si, 1.1 K, 4.8 Ca, 0.6 Ti, 0.1 Mn, 5.5 Fe. Hardness of coatings, phases and substrate are shown in Fig. 3 (measured by Buehler Micromet 2001 microhardness tester (HV0.05) and Indentec 5030 SKV Vickers hardness testing

Table 1. Parameters of the HVOF spraying process

Parameter	Value
Oxygen flow, l/min	920
Fuel flow (kerosene), l/min	0.36
Nitrogen flow, l/min	6.5
Combustion pressure, bar	7.1
Barrel length, inch	4
Spray distance, mm	380
Powder feed rate, g/min	152

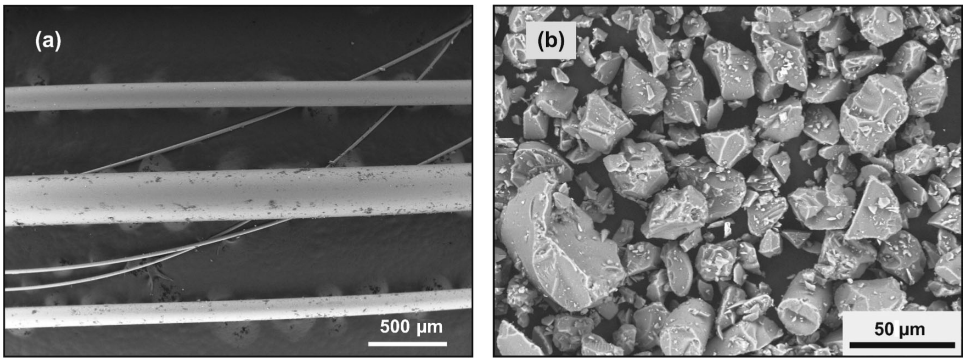

Fig. 1. SEM images: (a) original as-received basalt waste fibres of various thickness; (b) basalt powder after disintegrator milling.

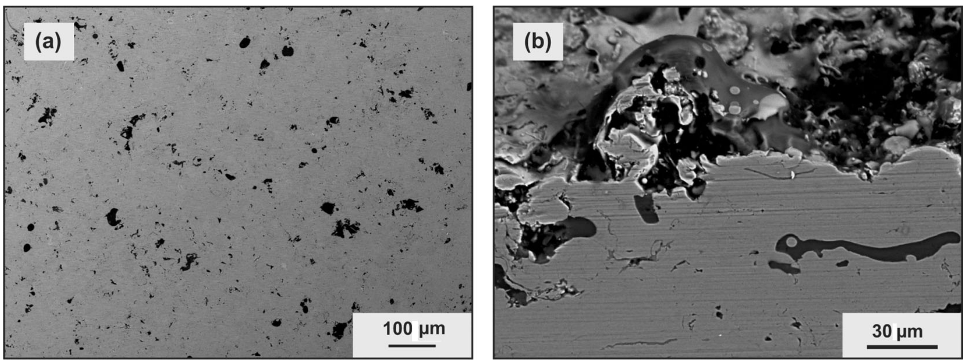

Fig. 2. SEM images of FeCrSiB-Basalt HVOF sprayed coating: (a) top view of the polished coating; (b) cross-section (top surface is not polished); dark areas indicate basalt.

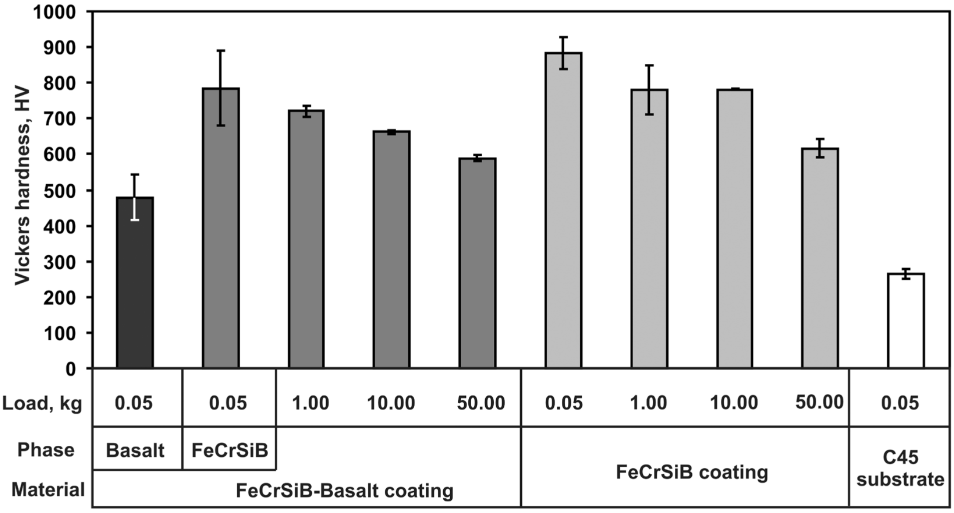

Fig. 3. Vickers hardness of HVOF sprayed coatings, basalt, steel phases and substrate measured after coating application.

machine (HV1, HV10 and HV50)). Images were obtained by Zeiss EVO MA15 scanning electron microscope equipped with Oxford Instruments INCA Energy System EDS.

2.2. Reciprocating sliding test conditions

Universal Micro Materials Tester (UMT-2) from CETR (Bruker) was applied for reciprocating sliding testing of coatings. Test conditions are shown in Table 2. The surface of the test sample is placed horizontally and the wear debris generated stay inside the wear track or are located around it.

Wear track profile was measured in the middle of its length by Mahr perthometer, PGK 120, in contact mode. Obtained area lost was multiplied by amplitude to get the volume of material lost.

Table 2. Reciprocal test conditions performed using UMT-2

Parameter	Description
Scheme	Ball-on-plate, plate is moving
Ball	Al_2O_3, HV1 = 1700, 3 mm in diameter
	Chrome steel EN 100Cr6 (AISI 52100), HV1 = 800, 3 and 10 mm in diameter
Plate	10 × 25 × 50 mm with HVOF sprayed coating applied
Amplitude	2 mm
Frequency (mean velocity)	1, 5, 10, 20 Hz (0.004, 0.020, 0.040, 0.080 m s^{-1})
Force against specimen	2.0, 4.9, 9.8, 78.4 N (0.2, 0.5, 1.0, 8.0 kg)
Atmosphere	Air, relative humidity 45 ± 10%. Temperature 25 ºC

2.3. Continuous sliding and three-body abrasion conditions

Continuous sliding with and without the abrasive was performed on Multi-functional Modular Tribosystem (MMTS) specially designed at TUT [8] (Table 3). This device enables one to measure the coefficient of friction (COF) of the ring-abrasive-block tribosystem. The ring is driven and the surface of the block undergoing wear is placed vertically that allows the wear debris to fall down once they are generated. During tests at elevated temperatures the sample (block) was heated. The temperature of the test surface before the test is controlled by an external contact thermocouple. After the beginning of the tests, the internal thermocouple is used for holding of the test temperature, taking into account the temperature drop. Load of 49N is sufficient to cause partial crushing of the abrasive [8]. Surface of the ring was cleaned by abrasive paper (silicon carbide, ISO/FEPA Grit P400) between tests.

2.4. Erosion testing conditions

Erosion tests were carried out using centrifugal accelerator CAK-5. Test conditions are summarized in Table 4. The device allows testing of 15 samples simultaneously in equal conditions [9].

Table 3. Sliding and 3-body abrasion test conditions performed using MMTS

Specification	Description	
	Continuous sliding	Three-body abrasion
Scheme	Block-on-Ring	
Ring	Ø 85 mm, breadth-10 mm, steel EN 10025 S355, HV10 = 230	
Block	10 × 25 × 50 mm with HVOF sprayed coating applied	
Circumferential velocity	0.25, 0.50, 1.00, 2.00 m s^{-1}	1.00 m s^{-1}
Linear abrasion	2670 m (10 000 rounds)	27 m (100 rounds)
Abrasive		SiO_2 with size of 0.2–0.3 mm, HV_1 = 1100, feed rate 300 g min^{-1}
Force against specimen	24.5 N (2.5 kg), 49 N (5 kg), 98 N (10 kg)	49 N (5 kg)
Atmosphere	Air, relative humidity 45 ±10%	
Temperature of test surface	25 ± 5, 300 ± 10, 500 ± 15 ºC	25 ± 5, 500 ± 15 ºC
Heating and cooling rates, holding time	Heating rate 15 °C min^{-1}, holding before test 20 min, cooling rate 25 °C min^{-1}	

Table 4. Erosion test conditions

Parameter	Description
Abrasive	SiO_2 with size of 0.2–0.3 mm, HV1 = 1100, 6 kg for one test
Impact velocity	25 and 50 m s^{-1}
Impact angle	30º
Atmosphere	Air, relative humidity 45 ± 10%, temperature 25 ºC

3. RESULTS

Results of the wear testing and SEM images of plain FeCrSiB coatings and of those with the addition of basalt are given in Figs 4 and 5. The wear rate in reciprocating and continuous sliding condition is of the same level. Higher hardness of alumina results in higher wear rates comparing to those obtained with

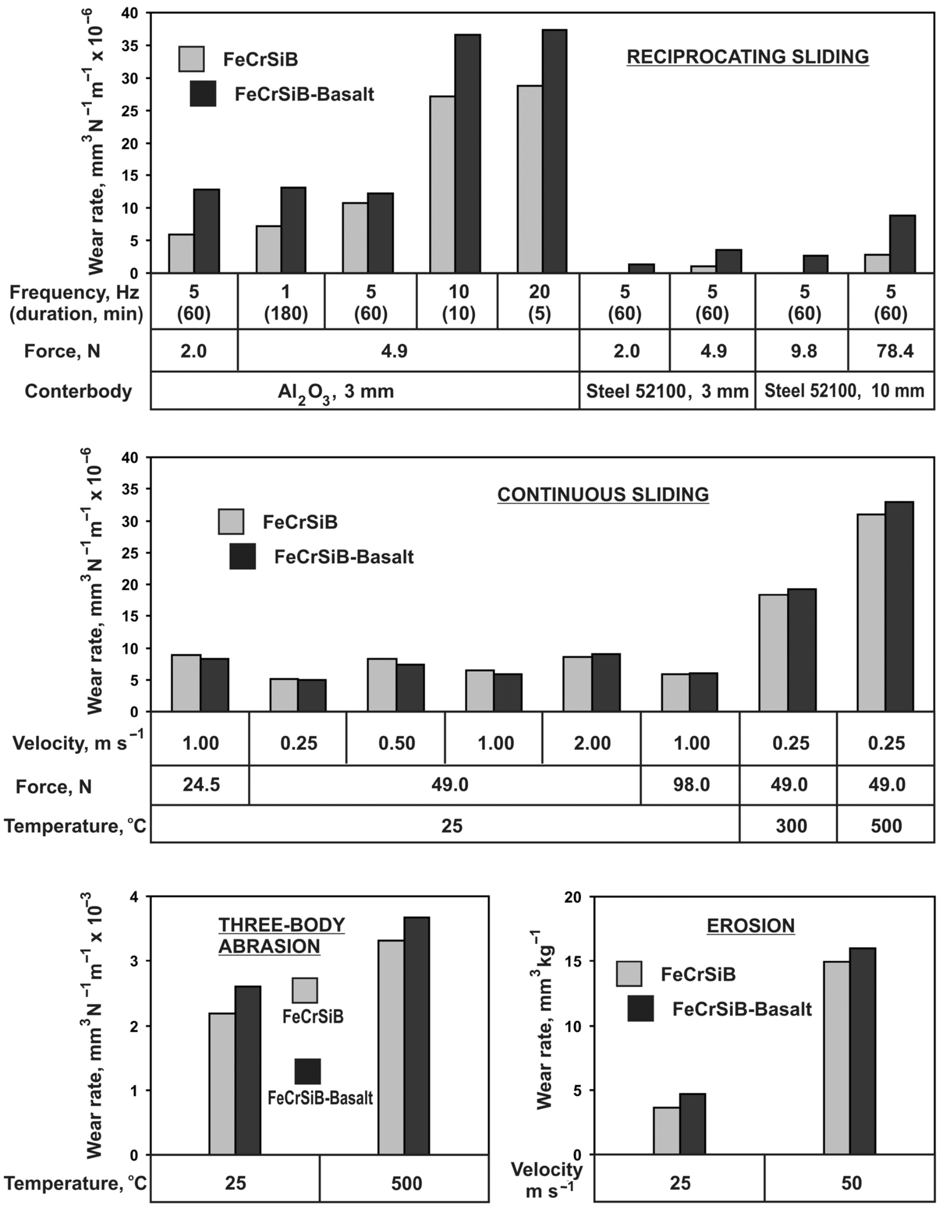

Fig. 4. Wear rates of coatings in various conditions.

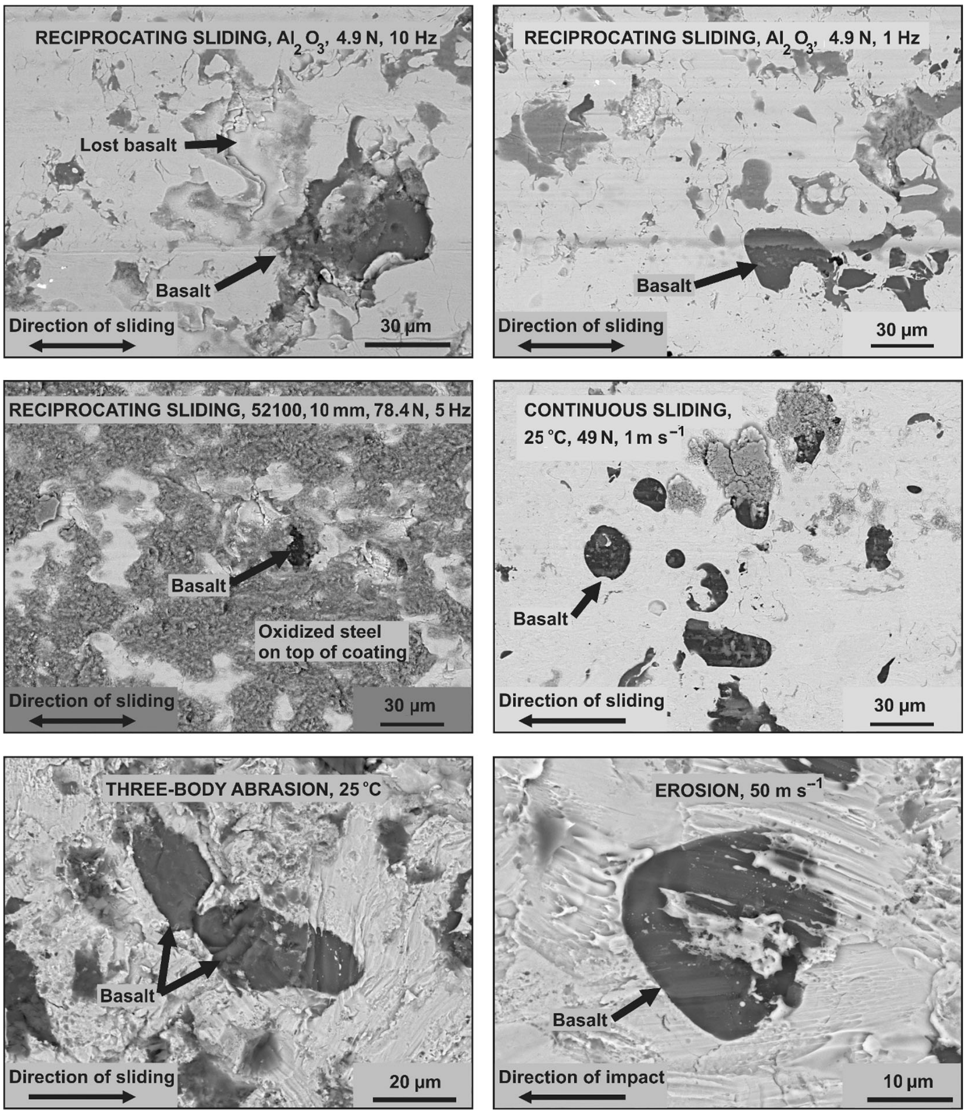

Fig. 5. Backscattered SEM images of the FeCrSiB-basalt coating after wear testing.

the chrome steel ball. During reciprocating sliding, the surface of the coatings is placed horizontally thus reducing the ability of wear debris to escape from wear track that reduce the wear rate for the tests with steel ball in contact. The highest wear in case of reciprocal wear was observed for short tests, carried out with high frequency of movements by hard aluminium balls. Basalt has the tendency to fall out at these frequencies (Fig. 5). High wear rate in the beginning is typical for non-conformal tests when the wear rate later decreases due to the reduction of the contact pressure, caused by the wear of one or both of the bodies in contact. Reciprocal tests result in the highest wear rate differentiation between coatings.

The most negative effect of basalt on the wear resistance of coatings takes place under the lowest load using alumina balls. Soft steel balls were not able to cause significant wear and even some transfer of steel to the coatings and its intensive oxidation was found (Fig. 5).

Only in continuous sliding mode the FeCrSiB coating with basalt was showing slightly better wear resistance than the plain one (Figs 4 and 5). Increased wear rates of both coatings were observed at higher temperatures that is explained by their softening.

Basalt addition has no measurable effect on COF of coatings. COF of both coatings was in the range of 0.50–1.10 and 0.50–0.75 for reciprocating and continuous sliding regimes, respectively. In three-body abrasive conditions the COF of the ring-abrasive-block tribosystems was in the range of 0.20–0.25 that is showing that the rolling of abrasive rather than ploughing and crushing takes place [8]. Rolling results in multiple impacting of the coatings surface, however, some sliding and ploughing also takes place (Fig. 5). The wear rates are much higher than in reciprocal and continuous sliding.

Wear rate of the basalt containing coating under erosive conditions is higher than that of plain coatings (Fig. 4). The difference in wear rates is reduced when coatings are tested at high velocity.

4. DISCUSSION

Basalt has lower hardness than the FeCrSiB coating (Fig. 3). Addition of basalt leads to the decrease in hardness of the composite coating that usually means a reduction in wear resistance as well. Adhesion between phases in a composite material is of paramount importance. Melting and high velocity of impact of basalt and FeCrSiB matrix powder during HVOF coating procedure enables to achieve a certain level of adhesion. However, basalt inclusions in the present composite are flake shaped and are easily broken during mechanical loading. That is why addition of basalt was favourable only in continuous sliding conditions with low velocity and low force (Figs 4 and 5). Wear process at low velocity generates less vibration. Almost no basalt is remaining during reciprocating sliding at a frequency of 10 Hz, while sliding at 1 Hz is milder (Fig. 5). It is also possible that basalt may act as a solid lubricant, thus reducing the adhesion between bodies in contact. Soft steel ring is favourable since it can embody some of the basalt rather than to generate extreme stresses that take place when wear particles get stuck between the alumina ball and coating. The concentration of basalt may be insufficient or it is required to coat the ring instead of the block. It was suggested in [10,11] to coat the body with largest area of surface in contact. Supply of solid lubricant is then sufficient to provide adequate lubrication. This is supported by the fact that if the continuous sliding test of FeCrSiB-basalt coating was repeated without cleaning the ring by abrasive paper then the wear rate of the coating was decreased.

It is required to make the shape of the basalt inclusions rather spherical than of flake shape by reducing the heat input during HVOF spray deposition and avoiding full melting. Also it is favourable to reduce the size of the basalt inclusions that facilitate the formation of mechanically mixed layer (MML) that is favourable in many cases [9,12]. This is supported by the fact that FeCrSiB-basalt coatings exhibited comparably good wear rates in erosive conditions under high velocity (Fig. 4) when formation of MML typically takes place in case of metal containing material. However, some precautions should be made to avoid burning of the fine basalt particles that is possible during the HVOF coating procedure. It should also be decided how to reduce the basalt losses during spraying due to the significant difference in the density of the powders.

5. CONCLUSIONS

1. Addition of macroparticles of basalt into FeCrSiB-alloy based HVOF sprayed coating has resulted in the reduction of wear resistance under most of the conditions where dynamic loading takes place.
2. Addition of basalt is favourable in continuous sliding conditions with low velocity, low force and with steel counterbody that may form surface layer enriched by basalt inclusions.
3. Shape and size of the basalt inclusions should be optimized to provide better resistance of FeCrSiB-basalt HVOF sprayed coatings against wear.

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REFERENCES

1. Singh, M., Modi, O.P., Dasgupta, R. and Jha A. K. High stress abrasive wear behaviour of aluminium alloy–granite particle composite. *Wear*, 1999, **233–235**, 455–461.
2. Czigány, T. Basalt fiber reinforced hybrid polymer composites. *Mater. Sci. Forum*, 2005, **473–474,** 59–66.
3. Cao, S., Liu, H., Ge, S. and Wu, G. Mechanical and tribological behaviors of UHMWPE composites filled with basalt fibers. *J. Reinf. Plast. Compos.*, 2011, **30**, 1–9.
4. Todic, A., Nedeljkovic, B., Cikara, D. and Ristovic, I. Particulate basalt–polymer composites characteristics investigation. *Mater. Des.*, 2011, **32**, 1677–1683.
5. Akinci, A., Ercenk, E., Yilmaz, S. and Sen, U. Slurry erosion behaviors of basalt filled low density polyethylene composites. *Mater. Des.*, 2011, **32**, 3106–3111.
6. Märkisches Werk, Crystal coat–an impermeable mineral-metal multiphase coating. http://www.mwh.de/Innovation/Innovations/MWH_CrystalCoat_CC_200.aspx (accessed online 05/05/2012).

7. Dietrich, M., Verlotski, V., Vaßen, R. and Stöver, D. Metal-glass based composites for novel TBC-systems. *Mat.-wiss. u. Werkstofftech.*, 2001, **32**, 669–672.
8. Antonov, M., Hussainova, I., Veinthal, R. and Pirso, J. Effect of temperature and load on three-body abrasion of cermets and steel. *Tribol. Int.*, 2012, **46**, 261–268.
9. Antonov, M., Hussainova, I., Pirso, J. and Volobujeva, O. Assessment of mechanically mixed layer developing during high temperature erosion of cermets. *Wear*, 2007, **263** (7–12), 878–886.
10. Michalczewski, R., Antonov, M., Vlad, M., Szczerek, M. and Hussainova, I. The assessment of the coated elements behaviour before and after scuffing under four-ball lubricated testing conditions. In *Proc. 2nd European Conference on Tribology ECOTRIB.* Pisa, Italy, 2009, 107–112.
11. Szczerek, M., Michalczewski, R. and Piekoszewski, W. The problems of application of PVD/CVD thin hard coatings for heavy-loaded machine components. In *Proc. ASME/STLE International Joint Tribology Conference.* Miami, Fl, 2008, 35–37.
12. Antonov, M. and Hussainova, I. Cermets surface transformation under erosive and abrasive wear. *Tribol. Int.*, 2010, **43**, 1566–1575.

Basaldi lisandi mõju kiirleekpihustusmeetodiga saadud FeCrSiB-pinde tribokarakteristikule

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Basaldi kasutamine komposiitmaterjalide valmistamiseks pakub huvi, kuna sel on rahuldavad mehaanilised omadused – kõvadus, madal tihedus ja suur korrosioonikindlus – ning see on odav. Kuna teraste ja basaldi sulamistemperatuurid on väga lähedased, siis on ainult kiirleekpihustusmeetodiga võimalik valmistada FeCrSiB-basaltpindeid, tugevaid, peaaegu poorivabu pindeid kõrgtemperatuurseteks rakendusteks. Basalti lisatakse soojuspaisumiskoefitsiendi sobitamiseks põhimaterjaliga. Leiti, et paljudes tribotingimustes (edasi-tagasi libisemine, abrasiivne erosioon) alandab basalt kulumiskindlust, kuna see on pehmem kui legeeritud teras. Samuti on basalt hapram ja selle osakesed on taldrikukujulised, ebasoodsa vormiga ning liiga suured. Ainult pidevas libisemisrežiimis mõjub basaldi lisamine positiivselt, kuna see võib toimida kui kõva määre.