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Sliding wear evaluation of hot isostatically pressed (HIPed) thermal spray cermet coatings

V. Stoica^a, R. Ahmed^{a,*}, T. Itsukaichi^b, S. Tobe^c

^a School of Engineering and Physical Sciences, James Naysmith Building, Heriot Watt University, Edinburgh EH144AS, UK
 ^b Fujimi Incorporated, 82-28 Kakamihigashi-machi-5, Kakamigahara, Gifu, Japan

^c Department of Mechanical Engineering, Ashikaga Institute of Technology, Ashikagashi, Tochigiken, Japan

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Abstract

The aim of this study was to investigate the potential of applying hot isostatic pressing (HIPing) as a post-treatment to thermally sprayed wear resistant cermet coatings. The relative performance of the as-sprayed and hot isostatically pressed functionally graded WC–NiCrBSi coatings was investigated in sliding wear conditions. Coatings were deposited using a high velocity oxy-fuel (HVOF)—JP-5000 system, and HIPed without any encapsulation at temperatures of 850 and 1200 °C. The influence of post-treatment temperature on the coating's wear resistance was thus investigated. Sliding wear tests were carried out using a high frequency reciprocating ball on plate rig using steel and ceramic balls under two different loads. Results are discussed in terms of microstructural investigations, phase transformations, mechanical properties, and residual stress investigations. The results indicated significant alteration of the coating microstructure, brought about by the coating post-treatment, particularly when carried out at the higher temperature of 1200 °C. As a consequence, developments in the coating mechanical properties took place that led to higher wear resistance of the HIPed coatings. © 2004 Elsevier B.V. All rights reserved.

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1. Introduction

Wear resistance of thermally sprayed coatings in sliding conditions has been a subject of numerous investigations over the last decades [1–3]. Different coating materials, spraying systems, and a number of tribological tests have been used in previous investigations. Based on their results, researchers proved that thermally sprayed coatings could be successfully applied to components used in different industries such as automotive, aerospace or mining. With the correct choice of the material for a particular application, using the developments in the process technology of thermal spraying, e.g. the latest high velocity oxy-fuel (HVOF) systems and the appropriate coating design, the coated components can excel in wear conditions.

Cermets are intensively used in thermal spraying industry for applications where wear resistance is required. Among them, the most attractive proved to be tungsten carbide based cermets. This is because tungsten carbide is known to have, beside the high hardness for which it was firstly chosen, a certain degree of ductility compared with the other carbides. This, in addition to the toughness provided by Co, CoCr, Ni or NiCrBSi matrix, forms not only very hard but also tough cermet system, making it suitable for numerous industrial applications.

The latest HVOF deposition systems are designed to optimise the velocity and temperature of the spray particles, hence decreasing the level of in-flight chemical reactions, and improving the bonding throughout the coating. A survey of published literature of such systems shows that the re-

^{*} Corresponding author. Tel.: +44 131 451 4722; fax: +44 131 451 3129. *E-mail address:* r.ahmed@hw.ac.uk (R. Ahmed).

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sulting coatings are more durable in wear applications due to their high hardness, density, low tensile or compressive residual stress when compared to the older HVOF or atmospheric plasma systems (APS).

Finally, the coating design, which accounts for the composition and the structure of the coating, plays a dominant role in controlling the tribo-mechanical properties of the coating. Thus, functionally graded depositions which bring a gradual change in both the composition and structure have been shown to alter the coating residual stress and thus to decrease the fatigue and fracture failures, leading to higher wear resistance.

However, in thermally spray coatings, even those sprayed by the latest liquid fuel JP-5000 systems, partial melting of the particles causes poor bonding at the interfaces between the unmelted and semi-melted particles, and phase composition changes due to in-flight chemical reactions. To a certain degree these problems are overcome by the availability of a number of post-treatments such as austempering/annealing, laser melting, and hot isostatic pressure (HIP). Promotion of cohesion between splats and adhesion between coating and substrate, phase transformation from amorphous to crystalline, generation of uniform compressive stress, and attenuation of coating anisotropy to obtain near homogeneous material properties are some of the desired post-treatment improvements, when a high degree of resistance to fatigue, impact loading, delamination, and corrosive/erosive/abrasive wear is required.

Previous studies have shown that the above-mentioned coating improvements may be achieved by the hot isostatic pressing (HIPing) post-treatment. The simultaneous application of high temperature and pressure promotes an attractive combination of material properties through changes in coating microstructure, beneficial phase transformations, and reduction in porosity levels [4–9]. It is anticipated that the advantages of the above-mentioned improvements will offset the cost of HIPing process in industrial applications.

This study addresses an investigation in which the wear performance of HIPed thermally sprayed WC–NiCrBSi coatings was investigated in sliding contact under different tribological conditions of load and counterface. Results are discussed in terms of microstructural investigations, phase transformation, mechanical properties, and residual stress investigations.

2. Experimental procedure

2.1. Material selection

The material selected for this study was WC-NiCrBSiFeC composite powder which was produced by the spray dried and sintered manufacturing route in two different compositions, i.e. WC-10 wt.%NiCrBSiFeC and WC-40 wt.%NiCrBSiFeC (Table 1). The rationale of choosing this powder over other wear resistant materials currently used in different industrial applications resides in a number of special properties brought to the system by the powder constituents, e.g. tungsten carbide brings the wear resistance, nickel brings excellent wettability which promotes good cohesion, chromium improves the tribo-mechanical properties, boron reduces the melting point, silicon increases self-fluxing properties, iron modifies the diffusion rates [10]. Hence, the material has very good sliding wear resistance, in two and three body wear by abrasive particles. Additionally, the above-mentioned properties provide very good protection against particle erosion and cavitation at temperatures up to 600 °C. Thus, components produced using this material are found in applications such as glass moulding plungers, hydro-turbines, and pump parts.

Moreover, taking into consideration the choice for the substrate, i.e. 440C stainless steel (see composition in Table 1) there is another benefit of using the NiCrBSiFeC. This is the promotion of a good bonding between the coating and the substrate, which could be further enhanced by the HIPing post-treatment. 440C stainless steel was chosen to be the substrate due to its superior properties over other steel grades, which help the substrate resists the harsh conditions of the HIPing treatment.

2.2. Coating production and post-treatment

A JP-5000 system was used to spray the powders on 31 mm diameter and 8 mm thick substrate discs. In a first stage, 100 μ m of WC–40 wt.%NiCrSiFeBC coating was deposited onto the substrate. This was followed by a 400 μ m layer of WC–10 wt.%NiCrSiFeBC, resulting in a 500 μ m functionally graded coating (Fig. 1). This coating was designed to bring a gradual change in the composition, particularly the use of higher amount of binder in the bottom layer

 Table 1

 Material selection for coating and substrate

Material selection for coating and substrate				
Powder	WC-10 wt.%NiCrSiBFeC	WC-40 wt.%NiCrSiBFeC		
Particle size distribution (µm)	- 45 + 15	- 45 + 20		
Apparent density (g/cm ³)	4.42	3.51		
WC grain size (µm)	3-4	3-4		
Powder manufacturing route	Agglomerated and sintered	Agglomerated and sintered		
Powder shape	Spherical	Spherical		
Alloy composition	Ni: Bal., Cr: 7.6%, Si: 3.6%, Fe: 2.4%, B: 1.6%, C: 0.25%	Ni: Bal., Cr: 7.6%, Si: 3.6%, Fe: 2.4%, B: 1.6%, C: 0.25%		
Substrate	440C Stainless steel			
Chemical composition (%)	Fe: Bal., C: 0.86–1.20, Mn: 1.0 max, Si: 1.0 max, P: 0.04 max, S: 0.03 max, Mo: 0.75 max, Cr: 16.0–18.0			



Fig. 1. Functionally graded coating system containing $100 \,\mu$ m layer of WC-40 wt.%NiCrSiBFeC and 400 μ m layer of WC-10 wt.%NiCrSiBFeC.

to provide an additional value to the coating wear resistance by decreasing the thermal and mechanical mismatch of properties.

In JP-5000 system, oxygen and kerosene were mixed in the combustion chamber (see parameters in Table 2), forming gases that accelerated the powder particles, depositing them onto the grit blasted and preheated 440C steel substrate. The post-treatment was carried out for 1 h at a constant pressure (150 MPa) in argon environment. The coatings were not encapsulated, decision mainly dictated by the economical and also technical incentives, and HIPed at 850 and 1200 °C.

2.3. Coating characterisation

2.3.1. Microstructural evaluation

The X-ray diffraction (XRD) analysis was used to quantify the microstructural modification for the as-sprayed and HIPed coatings. The diffraction patterns were obtained with a Siemens D500 diffractometer operating at 40 kV and 20 mA. Cu K α radiation was used and the samples were run at 2 θ from 10° to 90° with a step size of 0.02° (2 θ) and a time of 2 s/step. Spray powders and coating microstructures were evaluated by scanning electron microscopy using conventional imaging with secondary and backscattered electrons.

2.3.2. Microhardness and fracture toughness measurements

Microhardness measurements were carried out under a load of 300 g using a Vickers microhardness test machine on the coating surface and cross-section. Thirty measurements were performed on each coating surface and 150 on the cross-section, which were distributed in five lines of 30 measurements each, at 50 μ m from the substrate/coating interface on the substrate, 50, 150, 250, and 350 μ m, respectively from the substrate/coating interface on the coating cross-section.

The evaluation of coating fracture toughness was also carried out by the indentation method. Although several loads were used, fracture toughness could not be quantitatively evaluated [11] even at a load of 50 kg. Therefore, only a qualitative analysis was possible, which was based upon the

Table 2Spraying parametersSpray gunJP-5000Oxygen flow8931 l/minKerosene flow0.321 l/minSpraying distance380 mmSpraying rate50 g/min

optical microscope images showing the differences between the investigated coatings.

2.3.3. Elastic modulus measurements

The elastic modulus of all coating systems was determined by microindentation, with a Vickers indenter, using the universal hardness-measuring machine (Fischerscope HCU). A load of 500 mN was applied over 20 s and maintained for 5 s. Ten measurements were performed on each coating surface and 30 on each cross-section, which were distributed in three lines of 10 measurement points each, at 50, 150, and 250 μ m, respectively from the coating's surfaces. A distance of 100 μ m between all indentations was ensured to eliminate stress-field effect from nearby indentations.

Thus, the indentation modulus was determined with the help of the following relation:

$$Y_{HU} = \left(5.59h'_r \frac{\Delta h}{\Delta F}(h_{\max}) - 7.81 \times 10^{-7}\right)^{-1} [\text{GPa}] \quad (1)$$

where h'_r is the intersection point of the tangent at the load indentation depth curve for the maximum load with the indentation depth-axis (mm) and h/F (h_{max}) is the reciprocal slope of the tangent to the load indentation depth-curve at the maximum load (mm/N) [12]. Furthermore, the indentation modulus can be correlated to the Young's modulus (*E*) of the coating through:

$$E = Y_{HU}(1 - \nu^2) \tag{2}$$

where ν is the Poisson's ratio of the coating.

2.3.4. Residual stress measurements

Measurements of the residual stress on the surface and through depth of the as-sprayed and HIPed coatings were performed using X-ray radiations. Layers of 20 μ m were electropolished and then the residual stress was measured on the new exposed surface. The sin² ψ method was applied with eight ψ angles between 0° and 36°. The shift of the diffraction peak was recorded and the magnitude of the shift related to the magnitude of the residual stress through the slope of the 2θ -sin² ψ plot, according to the following relation:

$$\sigma = \frac{E}{1+\nu} \frac{\partial(\varepsilon)}{\partial(\sin^2\psi)} = -\frac{E\cot\theta_d}{2(1+\nu)} \frac{\partial(\theta)}{\partial(\sin^2\psi)}$$
$$= K \frac{\partial(\theta)}{\partial(\sin^2\psi)}$$
(3)

where *E* and *v* are the Young's modulus and the Poisson's ratio, respectively, ε is the residual strain, θ is the diffraction angle, θ_d is the diffraction angle in a stress free condition, ψ is the angle between the sample normal and diffraction-plane normal. *K* is the X-ray elastic constant and was measured using a conventional in situ four point bending test equipment subjected to a known load within the elastic range. The measured value of the *K* was—1975.13 MPa. The last term of the equation is the slope of the best fitted straight line of the 2θ -sin² ψ plot.



Fig. 2. High frequency reciprocating ball on plate rig.

2.4. Tribological testing

Sliding wear tests were carried out using a reciprocating ball-on-plate apparatus, instrumented to measure the frictional force via a load cell (Fig. 2). As it can be appreciated from the figure, the upper ball bearing the normal load is stationary while the coated disc has a reciprocated motion. The sliding speed was 0.037 m/s at the centre of the wear scar. The type and properties of the balls are listed in Table 3. The coated specimens were ground and polished to produce a surface roughness (R_a) of 0.04 µm. Before each test, the coatings and balls were ultrasonically cleaned in acetone for 5 min to remove any contaminants and grease and then dried in air.

The tests were performed under two normal loads (12 and 22 N) in unlubricated contact conditions at ambient temperature and humidity. The contact stresses at the beginning of the tests, corresponding to the loads applied by the steel balls were approximately 1 GPa for 12 N load and 1.2 GPa for 22 N load, each of these values varying slightly depending on the type of the coating. When the ceramic balls were used, the contact stresses were approximately 1.18 GPa and 1.2 GPa corresponding to the load of 12 and 22 N, respectively. The repeatability of wear results was considered by repeating each test three times, and averaged values are presented in the next section.

The coating wear scars were examined using Zygo New View 5000 interferometer, which provided the volume loss of the material for each wear scar. The volume loss of the ball was evaluated using an optical microscope, which allowed the precise measurement of the ball wear scar diameter. Moreover, using the diameter of the sphere segment removed during the test, the ball volume loss (*V*) was calculated as follows: $V = \pi H^2(3R - H)/3$, where $H = R - \sqrt{R^2 - r^2}$ and "*R*" and "*r*" are the ball radius and ball-wear-scar radius, respectively. The surface morphology of the coating wear scars was analysed with SEM equipped with EDX.

Table 3

The properties of the balls used as counterface in sliding tests

	-	
	440C Steel	Si ₃ N ₄ ceramic
Diameter (mm)	12.7	12.7
Density (kg/m ³)	7769	3165
Weight (g)	8	3
Average roughness R_a (µm)	0.015	0.013
Hardness (HV0.1) (kg/mm ²)	820	1580

3. Experimental results

3.1. Microstructural identification

3.1.1. X-ray diffraction analysis

The XRD patterns of the spray powder, as-sprayed and HIPed coatings are presented in Fig. 3. Fig. 3a shows the phase composition of agglomerated and sintered WC-10 wt.%NiCrSiBFeC powders. As expected high amounts of mono tungsten carbide (WC) and certain quantity of metallic nickel and chromium can be observed in the powder spectra. However, beside these phases, some amounts of chromium carbides (Cr_xC_y) are also seen, which were brought about by the powder manufacturing process.

Deposition of these powders under the conditions listed in Table 2 led to the formation of secondary phase tungsten carbide (W₂C) and metallic tungsten (W). Further evidence of coating decomposition can be seen by the peak broadening between 42.7° and 44.7°, which indicates the presence of amorphous/nanocrystalline phase.

The post-treatment carried out at 850 °C produces visible changes in the coating phase composition (Fig. 3c). Thus, higher amounts of primary tungsten carbide, less amounts of secondary phase (W₂C) and metallic tungsten (W) are noticed. The occurrence of two additional phases, complex carbide containing Ni (Ni₂W₄C) and cromium carbide $Cr_{23}C_6$ is also observed.

When the post-treatment is carried out at a temperature of 1200 °C, significant phase transformations take place compared to as-sprayed coating (Fig. 3d). The higher temperature allows the formation of additional phases which are not present in the coatings HIPed at 850 °C. Thus, beside an increase in the existent nickel-containing carbide (Ni₂W₄C) and chromium carbide (Cr₂₃C₆), new phases such as iron tungsten carbide (FeW₃C) and chromium carbide (Gr₃C₂, Cr₇C₃). Moreover reactions between Ni and B or N and Cr take place and thus phases such as Ni₄B₃, NiB₁₂, Cr₃Ni₂ form.

3.1.2. SEM observations

Fig. 4 illustrates the characteristic spherical shape of the agglomerated and sintered powders used for coating deposition. The size of the powder particles measured via image analysis on this micrograph ranges between 30 and 60 μ m with an average at around 45 μ m.

Figs. 5–7 show SEM micrographs taken on the crosssection of the as-sprayed and HIPed coatings. Figs. 6d and 7d exhibit the interface between the HIPed coatings and the substrate. Contrary to the as-sprayed coating shown in Fig. 5d, an additional layer, slighly brighter than the matrix or the substrate, occurred, in both cases, along the interface. This layer is approximately 5 μ m for the coating HIPed at 850 °C and about 25 μ m for the coating HIPed at 1200 °C.

During the examination of the coating layers no obvious difference in the shape or size of WC grains was noticed between the as-sprayed and the coating HIPed at 850 °C as



Fig. 3. XRD spectra of (a) WC-10 wt.% NiCrSiBFeC powder and top layer of (b) as-sprayed, (c) coating HIPed at 850 °C, and (d) coating HIPed at 1200 °C.



Fig. 4. SEM micrograph of the starting WC-10 wt.%NiCrSiBFeC powder.

shown in Figs. 5 and 6. However, the micrograph of the coating HIPed at 1200 °C shows significant changes in the structure of the coating. Throughout the coating, within both layers, the carbides underwent a transformation in the shape from rounded to prismatic. Moreover there was an obvious difference between the two coating layers. This is the presence of carbide agglomerates in the layer which has a higher level of binder. Hence, the carbide grains are dragged one to another and they form closely-packed islands which, as a result, increases the areas of matrix which do not contain carbide grains (Fig. 7c).

Finally, a closer look at the interface between the bottom and the upper layers of the coating HIPed at the temperature of $1200 \,^{\circ}\text{C}$ reveals the existence of another layer of a thickness of approximately $15 \,\mu\text{m}$. The structure of this intermediar layer is rather similar to that of the bottom layer of the coating HIPed at $850 \,^{\circ}\text{C}$ although the grains have the angular shape characteristic to the coating HIPed at $1200 \,^{\circ}\text{C}$ (see Figs. 6c and 7b).

In order to avoid misinterpreting any feature which might have arose on the coating cross-section (especially structure and pores) due to mechanical deformation during the cutting process or due to grinding or polishing [13–15], the coatings were immersed in liquid nitrogen for 3 h and then fractured. Fig. 8 shows the scanning electron micrographs of the fractured coatings. A number of features can be appreciated when micro-analysing the as-sprayed coating. Lamellar cracks, indicated by arrows in Fig. 8a, appear to be distributed over the investigated regions of the coating. Black spots, signed "A" on the same figure, whose diameters do not exceed approximately 1.5 µm can also be seen on the fractured as-sprayed coating. A different aspect can be noticed on the fractured HIPed coatings. Also, the surfaces of both HIPed coatings were much rougher than that of the as-sprayed coating. No microcracks are observed but the dark spots, intentionally shown in Fig. 8c at a slightly higher magnification for more clarity, suggest that the level of porosity on these coatings is slightly higher than that on the coating HIPed at 850 °C. It is believed that upon application of pressure and temperature,



Fig. 5. SEM micrographs of the as-sprayed coating: (a) general view, (b) interface between top and bottom layers, (c) bottom layer, and (d) interface between the coating and the substrate.



Fig. 6. SEM micrographs of the coating HIPed at 850 °C: (a) general view, (b) interface between top and bottom layers, (c) bottom layer, and (d) interface between the coating and the substrate.



Fig. 7. SEM micrographs of the coating HIPed at $1200 \,^{\circ}$ C: (a) general view, (b) interface between top and bottom layers, (c) bottom layer, and (d) interface between the coating and the substrate.



Fig. 8. SEM micrographs on cross-section of cryogenic fractured: (a) as-sprayed (the arrows indicate cracking, "A" indicates pores), (b) HIPed at 850 °C, and (c) HIPed at 1200 °C coatings (upper layer).

the small-in-size pores of the HIPed coatings arise from the fragmentation of the lamellar cracks and/or collapsing of the bigger pores already seen in the as-sprayed coatings. However, lamellar cracks completely disappear at higher HIPing temperature of 1200 °C.

3.2. Mechanical properties measurement

3.2.1. Microhardness measurements

The microhardness values for all the coatings that were tested are shown in Fig. 9. The measurements taken on the substrate cross-section in the vicinity of interface with the coating shows an increase of the substrate microhardness when the coated discs were subjected to the temperature and pressure of HIPing process. As can be appreciated from this figure, the microhardness of the substrate in that particular location is very sensitive to the HIPing temperature. Thus, the microhardness increased from 400HV₃₀₀ to approximately 600HV₃₀₀ when the temperature is raised from 850 to 1200 °C. An opposite trend was seen on the results of the measurements carried out at 50 μ m on the coating cross-section. At this location, which is approximately in the middle of WC–40 wt.%NiCrSiBFeC layer, the as-sprayed coatings have the highest microhardness, 1134HV₃₀₀, fol-

lowed by the coating HIPed at 850 °C with 971HV₃₀₀ and the coating HIPed at 1200 °C with 846HV₃₀₀. The rest of the cross-section measurements were performed on the WC-10 wt.%NiCrSiBFeC layer. The results show, as expected, higher microhardness compared with the first layer. For the entire thickness of this layer, the HIPed coatings have a higher microhardness than the as-sprayed coatings. An increase of about $100HV_{300}$ is brought about by treating the coating at 850 °C, while increasing the temperature of the post-treatment at 1200 °C increases the microhardness from around 1100 up to approximately 1250HV₃₀₀. These values were calculated as average on the entire thickness of the bottom layer. The microhardness measured on the coating surface keeps the same ascending trend from the as-sprayed to the coating HIPed at 1200 °C. However, the results show that on the surface, HIPing at 850 °C has minor influence on the coating microhardness, a rather insignificant increase from $1005HV_{300}$ to $1015HV_{300}$ being noticed for coatings HIPed at 1200 °C.

3.2.2. Fracture toughness measurements

In order to determine coating's toughness, the coatings were indented using a Vickers macrohardness apparatus. Initially the indentations were performed under 5 kg. However,



Fig. 9. Variation of coating microhardness with the distance from the coating/substrate interface. () as-sprayed, () HIPed at 850 °C, () HIPed at 1200 °C.

the crack length of all coatings could not meet the criteria c > 2a, required for the determination of numerical value of fracture toughness (see ref. [11] for details). The load was further increased but, even at a load of 50 kg, quantitative

analysis of the fracture toughness could not be performed. Therefore, using the micrographs taken with an optical microscope (Fig. 10), only qualitative observations on the coating fracture mode could be made.



Fig. 10. Fracture toughness indentations on (a) as-sprayed, (b) HIPed at 850 °C, and (c) HIPed at 1200 °C coatings (all coating tested at 50 kg).



Fig. 11. Variation of coating elasticity with the distance from the coating surface.

The micrograph of the as-sprayed coating (Fig. 10a) shows a net of cracks spread around the indentation. The cracks originated not only from the corners but also from the each side of the indentation, propagating randomly in the surroundings. Differing, the indentations of the coating HIPed at 850 °C exhibit less cracks, limited only in the close vicinity of the indentations. The coatings HIPed at a temperature of 1200 °C produced tougher coatings, conclusion drawn after analysing all the indented coatings. As Fig. 10c reveals, no cracks were produced indenting the coatings with a load as high as 50 kg.

3.2.3. Indentation modulus measurements

The results of the coating elasticity measurements are shown in Fig. 11. This graph indicates the difference in the elastic modulus between the as-sprayed and the post-treated coating and the influence of the HIPing temperature on the coating elasticity. The results show higher coating elastic modulus brought about by the application of the coating posttreatment. Thus, an increase of almost 70% is noticed after HIPing the coatings at 850 °C. Moreover, following Fig. 11, it can be appreciated that an increase in the post-treatment temperature leads to higher coating elasticity. The results also show slightly higher anisotropy for the coating HIPed at 1200 °C whose modulus gradient is the highest when compared to the other two coatings.

3.3. Residual stress measurements

The results of residual stress measurements are summarized in Fig. 12, which describes how the residual stress changes when the coatings are post-treated by the HIPing process. Firstly, as expected the residual stress of all coatings is compressive. Secondly, it is observed that HIPing the coatings at 850 °C leads to an increase in compressive stress of about 9%, from 780 MPa to approximately 850 MPa. HIPing the coatings at 1200 °C further increases the stress to 990 MPa. All these values were calculated by averaging the residual stress over the coating thickness. In terms of stress gradient along the coating thickness, no difference is noted between the stress distributions of the as-sprayed and HIPed at 850 °C coatings. Both these coatings exhibit in some regions stress variations of almost 100 MPa across a thickness of 20 μ m. On the other hand, the coating HIPed at 1200 °C has a maximum variation of approximately 50 MPa, which is spread over the first 60 μ m.

3.4. Tribological testing

3.4.1. Friction behaviour

The friction coefficients of all tested couples are presented in Figs. 13 and 14. Fig. 13a and b shows the friction behaviour of the couples tested using the lower load. The online measurements indicated higher frictional force for all coatings tested against steel balls compared with coating–ceramic couples. Correspondingly, as shown in the above-mentioned figures, the friction coefficients are higher, with averaged values of 1.13 and 1.03, respectively.

When the balls applies 22 N on the coating, contrary to the above case, the frictional force resulted from coating–steel contact is smaller than that produced by the coating–ceramic couples. This is shown in Fig. 14a and b where the friction coefficient of the couples involving steel have a value of 0.87 compared with 0.94 obtained rubbing the ceramic balls on the coatings. Summarizing this steel–ceramic comparison, regardless of the type of coating used, at low load (12 N) steel produces higher friction while, at high load (22 N), higher friction is given by the ceramic.



Distance from the coating surface (µm)

Fig. 12. Variation of coating residual stress with the distance from the surface.

Comparing Fig. 13a with 14a and thus, the friction coefficients produced sliding steel balls over the coatings, it is observed that the tests carried out at a load of 22 N produces less friction than those using lower load. The average value of friction coefficient decreases from 1.13 to 0.87 with increasing load from 12 to 22 N. The same trend is given by the graphs in Figs. 13b and 14b. Thus, the average friction coefficient of the coating–ceramic couples is 1.03 and 0.94 for low and high load, respectively.

Hence, regardless the type of ball used in the test, increasing the load produces a decrease in the friction coefficient. This is bearing in mind that only two different loads were used. The friction coefficient values presented above were calculated as average of the friction coefficients of the assprayed and HIPed coatings over the total test time after the running-in period.

No clear difference between the as-sprayed and HIPed coatings when tested against either steel or ceramic balls is observed. Typical friction behaviour of the sliding tests is found for all the couples tested: the running-in stage which in almost all cases last around 140 mins, followed by the stabilisation stage characterised by a rather random oscilation of the friction coefficients around the average value.

3.4.2. Sliding wear

A Zygo interferometer was used to measure the volume loss of the coating material. The results for all coatings involved in the testing are shown in Fig. 15. This provides the relative performance comparison of the as-sprayed and HIPed coatings. Furthermore, the volume loss of the entire system was determined adding the volume loss of the balls to that of the coatings and the results were compiled in Fig. 16.

The general trend, regardless of the corresponding counterface or the load used, is that the HIPed coatings lost less material than the as-sprayed ones. This essential finding is shown in Fig. 15. Although no significant improvement in the wear resistance of the coatings HIPed at 850 °C can be noticed, the coating HIPed at 1200 °C loses almost half the material lost by the as-sprayed coatings. In this case, the performance of the coating HIPed at 850 °C is rather an exception. The rest of the couples show almost twice the wear resistance of the coatings HIPed at 850 °C, followed closely by the coating HIPed at 1200 °C.

Adding the volume loss of the balls gives the total volume loss of the tested systems. Fig. 16 confirms furthermore the trends shown in the previous figure. Here the amount of the ball material lost during testing accentuates the difference between the as-sprayed and the HIPed coatings. Thus, in most cases, the ball which rubs on the as-sprayed coatings produced double or even more system material loss. This behaviour is consistent at both loads and also with steel and ceramic balls, indicating the consistent trend of improved wear performance over the range of tribological conditions considered in this investigation.

The interferometric plots also provided three-dimensional plots of the middle section of coating wear scars. Some of them, those which were tested against ceramic balls under 22 N are presented in Fig. 17. The particularities of each coating wear scar such as width or depth can be observed from this figure. As a general trend, the scars on the as-sprayed coatings are significantly wider than those on the HIPed coatings.



Fig. 13. Friction coefficient of the as-sprayed and HIPed coatings tested vs. (a) 440C steel and (b) Si_3N_4 balls (load = 12 N).

In most cases the wear scars on the coating HIPed at $1200 \,^{\circ}$ C is the narrowest of them. In terms of wear track depth, the as-sprayed coatings are again the worst, being the deepest regardless the load or ball used. Quantifying for the couples presented in Fig. 17, the wear track on the as-sprayed coating has 5.8 µm depth and 1.6 µm width. Compared to this, the depth of the HIPed coatings wear scars is less than half, 2.25 µm and 1.8 µm, respectively. Same trend was seen when comparing the width of these coatings although the difference between the coatings is less. Thus, the width of the coating HIPed at 850 °C is 1.4 µm whilst that of the coating HIPed at 1200 °C is 1.38 µm.

Fig. 18 summarizes the post-test investigations of the wear tracks of the as-sprayed and HIPed coatings which slid against steel and ceramic balls, whereas the debris resulting from sliding wear tests are shown in Fig. 19. Fig. 20 shows the EDX analysis within the wear track of the as-sprayed coatings.

4. Discussion

Previous study [16] by authors proved that HIPing posttreatment at temperature of $850 \degree C$ for 1 h can be successfully



Fig. 14. Friction coefficient of the as-sprayed and HIPed coatings tested vs. (a) 440C steel and (b) Si_3N_4 balls (load = 22 N).

applied on encapsulated thermally sprayed WC–Co coatings. In the current research, the authors investigated the potential of combining special properties of functionally graded coatings, with the properties of WC–NiCrBSi material to enhace the beneficial effect that HIPing post-treatment has on wear resistant coatings. Moreover, the HIPing conditions were specially chosen to avoid cracking of coatings during heating and cooling, and to decrease the cost of the process using unencapsulated coatings. The post-treatment of these coatings was performed at 850 °C and also at 1200 °C to investigate the effect of a higher HIPing temperature on the capacity of these specially designed coatings to withstand harsh conditions in wear applications.

A detailed analysis of the microstructure of the HIPed coatings was performed, the mechanical properties and residual stresses of the HIPed coatings was compared to those of the as-sprayed ones and the coatings were tested in sliding conditions.

4.1. Coating microstructure

An analysis of chemical composition by XRD diffraction revealed a number of phase transformations which take place during the coating post-treatment. Secondary phases of tungsten carbide (W_2C) and the metallic tungsten which are known to contribute towards the coating's brittleness be-



Fig. 15. Coating volume loss (mm³).

come unstable at temperatures above $850 \,^{\circ}$ C. It is believe that reactions of these phases with the existent nickel or iron from the matrix led to the formation of additional complex carbides (Ni₂W₄C, FeW₃C). The increased intensity of the WC peaks suggests that some secondary phase also converted to WC, conversion facilitated by the carbon which existed in the matrix (Table 1). Thus, although there are still some brittle secondary phases of tungsten carbide in the coatings HIPed at 850 °C, they are less than in the as-sprayed coating. Therefore, these post-treated coatings can be expected to be tougher. Moreover once the complex carbides formed, they should bring extra hardness to the coatings HIPed at 850 °C. Their influence on toughness can also be appreciated from Fig. 10.

The increased number of new phases in the coatings HIPed at 1200 °C indicates more reaction activity with increasing the HIPing temperature. Thus, as a consequence, brittle secondary tungsten carbide and metallic tungsten phases which were seen in small amounts in the coating HIPed at 850 °C were eliminated. Except these brittle phases, the rest of the phases existent in the coating HIPed at 850 °C also formed in the coatings HIPed at 1200 °C. Due to higher temperature, additional reactions increased the number of complex carbides such as Ni₂W₄C, chromium carbides Cr_xC_y , and also led to the formation of new carbides such as FeW₃C or products of nickel and chromium or nickel and boron. Bearing all these transformations in mind, the changes in mechanical properties of the HIPed coatings can be explained. Thus, the absence of secondary tungsten carbide phase decreased the coating brittleness while the formation of additional complex carbides led to higher coating hardness. Moreover, a survey of the literature also shows that nickel borides or chromium nickel phases would increase the hardness of the material whose constituents they are [17–19].

Microanalysing the HIPed coatings under scanning electron microscope, marked characteristics of the structure of the HIPed coatings particulary those HIPed at 1200 °C are found, and these differences from the structure of the as-sprayed coating can be appreciated by studying the coating layers. Micrographs taken on the vicinity of the coating/substrate interface of both HIPed coatings reveal the occurrence of an intermediar layer along the interface while the initial interface disappeared (e.g. Fig. 7d). Elemental analysis within this region indicates the presence of high amounts of Ni, suggesting that diffusion-controlled processes took place between the substrate and coating layer, from the coating towards the substrate, leading to the strengthening of the bonding between the two parts and correspondingly, the elimination of the initial interface. An additional effect of the diffusion of the binder in the substrate is expected to be the hardnening of the substrate throughout the additionally-created layer. As expected, a greater extent of this metallurgical bonding occurred during HIPing at 1200 °C, which was confirmed by the difference of the thickness of the new diffused layer, and also in the substrate hardness near the coating/substrate interface.

As illustrated in the Figs. 5b and d, and 6b and d, no clear evidence was observed of any effect of the post-treatment carried out at 850 °C on the size and shape of carbide grains. Thus, both the as-sprayed and the coating HIPed at 850 °C have approximately the same characteristics. This was valid for both the upper and bottom coating layers. Hence, 850 °C proved to be lower than the temperature needed to cause substantial changes thoughout the coating microstructure.

Contrary to above, the post-treatment carried out at 1200 °C produced appreciable changes (Fig. 7). Firstly, noticeable throughout the coating is the tendency of the carbide grains to become prismatic. Moreover, under the pressure of the post-treatment a rearrangement of the grains is observed and thus a shift from a relatively homogenous distribution of



Fig. 16. Total volume loss (mm³) of (a) coating-steel and (b) coating-ceramic systems.

carbide grains of the coating HIPed at 850 °C to the formation of islands of grains takes place. However this aggregation of the carbides brings about an increase in the size of binder spaces. Since this is particulary evident in the bottom layer of the coating HIPed at 1200 °C, the carbide agglomeration is related to the level of binder in the coating. With the HIPing temperature higher than the eutectic, the solubility of carbides in the binder is greatly enhanced. This decreases the activation energy for atomic movement across the carbide boundaries leading to greater flexibility of the carbide interfaces [20]. The movement of the carbide interfaces ceases when low energy prismatic carbide interfaces form. Close-packing of carbides takes place and thus the contact between different carbides increased. Furthermore, another process might take over which leads to a coarsening of the grains. This may be the coalescence of the carbide grains as described by Ryoo et al. [21]. Although the adsorbtion of additional atoms on the surface of the prism-shape carbide would increase the surface energy, nevertheless, the micrographs show intimate contact between the certain grains to the extent that the boundary between them cannot be easily distinguished. Thus, provided that an angular relationship between two grains is satisfied [21], they can easily coalesce forming larger prismatic grain. However, there are situations when the corsening of the grains is possible only with an influx of C atoms, which decreases the probability of coalescing, whilst sometimes the orieta-



Fig. 17. Interferometric plots of the (a) as-sprayed, (b) HIPed at 850 $^{\circ}$ C, and (c) HIPed at 1200 $^{\circ}$ C coating wear scars produced by ceramic balls (load = 22 N).

tion relationship is totally unfavorable and the coalescence impossible.

Another feature specific to the coatings HIPed at $1200 \,^{\circ}\text{C}$ is the layer which occurred between the bottom and the upper layers. Since its structure resembles the bottom layers of the coating HIPed at $850 \,^{\circ}\text{C}$, it is believed that a longer HIP-ing time would have transformed the structure of the coating HIPed at $1200 \,^{\circ}\text{C}$ over the entire thickness of the bottom layer.

4.2. Mechanical properties

Mechanical properties measurements confirmed the assumptions made throughout the previous section on the effect of microstructure on the coating hardness or toughness.

Thus, the gradual increase of the microhardness with HIPing temperature is brought about by the occurrence of new hard phases throughout the upper coating layer. The analysis of the phase composition also shows that an elimination of brittle phases takes place during the HIPing process, and is enhanced with increasing the temperature. Although only qualitative, the fracture toughness results show consistent behaviour confirming the above. Hence, there is an increase of the potential of the upper coating layer to withstand higher loads without fracturing with the temperature of the posttreatment. However, a simultaneous increase in the hardness and the toughness will not be reasonable without an improvement of the elasticity of the upper coating layer. The modulus results taken on the surface and throughout the cross-section of this layer of both HIPed coatings validated this assumption. Hence, it is believed that the transformations in the phase composition of the coatings during the post-treatment set up new relatively stronger bonds at 850 °C, which is reflected by the substantial increase in the elasticity of the HIPed coatings compared with the as-sprayed ones. However, the modulus results of the coating HIPed at 1200 °C shows even higher values. Although the coating HIPed at 1200 °C has higher modulus, the coating treated at 850 °C has lower modulus gradient, and hence almost no anisotropy. The source of the anisotropy might be the porosity level of the coatings HIPed at 1200 °C (Fig. 8c), which influences the measurements taken at low load of 500 mN.

On the other hand, for the bottom layer, the particular coating structure is responsible for the decrease in the microhardness of the coating HIPed at 1200 °C. The transformation from the relatively homogeneous carbides distribution to the formation of aggregates produced larger spaces of binder which were influential in microhardness measurements due to the corresponding decrease of the number of carbides to sustain the load. Thus, it is thought that the plastic deformation component during the indentation is responsible for the difference in the microhardness results.

The results of the microhardness measurements taken on the substrate in the close vecinity of coating/substrate interface show an increase in the hardness of the substrate in this particular location. The alteration of the substrate composition as a result of nickel diffusion which was mentioned in the previous section is responsible for the enhancement of the substrate hardness. Regardless of the temperature of HIPing, all treated systems had an increase of substrate hardness which is nevertheless increasing as the HIPing temperature increases.

4.3. Residual stress measurements

In terms of residual stress, the HIPing process builds up compressive residual stresses within the coating, thus increasing the stress level already existing in the as-sprayed coatings (Fig. 12). It is believed that, during the HIPing process, the residual stress profile undergoes a number of changes corresponding to the influence of the pressure and temperature



Fig. 18. SEM micrographs within the wear scar of as-sprayed, HIPed at $850 \,^{\circ}$ C and HIPed at $1200 \,^{\circ}$ C coatings tested against steel: (a), (b), (c), and ceramic (d), (e), (f) under a load of $12 \,$ N. ("A" indicates oxide layers and the arrows indicate cracking of the oxides under the load).

during the post-treatment. Considering the result of the measurements, it can be appreciated that the application of the pressure produces enough compressive stresses to overcome the tensile stresses which resulted from the cooling stage. The temperature of the HIPing process doesn't significantely influence the level of the stresses, the HIPed coatings having about the same averaged value calculated over the entire coating thickness. However, Fig. 12 shows an evident difference between these coatings. This is the gradient of the stress value over the coating thickness. Thus, the coating HIPed at 1200 °C has less variation of residual stress throughout the coating thickness, indicating that stress concentrations are less likely under mechanical loading.

4.4. Tribological testing

The total wear of the coating-ball system is influenced by several main mechanisms: microcraking of the coating surface, oxidation at the contact asperities, abrasive wear in two and three body. Although the contribution of these wear



Fig. 19. Debris formed by sliding the as-sprayed, HIPed at 850 $^{\circ}$ C and HIPed at 1200 $^{\circ}$ C coatings against steel: (a), (b), (c), and ceramic balls (d), (e), (f) (load = 12 N).

mechanisms cannot be treated independently, it is convenient for a better understanding to discuss them separately.

Regardless of the tribological conditions microcracking is a wear mechanism which occurred only in the tests which involved the as-sprayed coatings. Thus, the micrographs taken on the as-sprayed coatings exhibit microcracks randomly dispersed over the wear track. They are ascribed to the relatively higher brittleness of these coatings compared to the HIPed ones. The cracks nucleate at the surface of the coating within the brittle phases and propagate along the boundaries of WC grains (Fig. 17a) throughout the contact area, preferentially perpendicular to the sliding direction. They influence both the friction and wear of the system. Thus, as energy dispersive mechanism, microcracking causes an increase in friction. The crack edges also cause the material of the ball to be removed and are themselves preferred location for the removal of coating material. Moreover propagating the cracks form nets, leading to localised weakening of the



Fig. 20. EDX analysis within the wear scar of the as-sprayed coating tested against (a) steel and (b) ceramic ball (load = 12 N).

coating surface and, subsequently to the delamination of the coating.

Transient flash temperatures at the asperities contact lead to oxidation throughout the contact area. Hence, layers of dark phase (Fig. 18), confirmed by EDX to have high proportion of iron and oxygen, and tungsten (Fig. 20a and b) randomly covered the coatings tested against steel and ceramic. This suggests the presence of FeO in the first case (steel ball) and WO₃ in the later (ceramic ball). Since smearing of the oxide layer was noticed under the SEM, it is suggested that further sliding causes the spreading of the oxide layers over the coating. They also deposit into the surface pits providing reservoirs for further system solid lubrication, decreasing the friction coefficient and thus avoiding adhesive wear. Subsequent formation and removal of these oxide layers is a part of this process which last for the entire duration of the wear tests. Also called the oxidational wear, this process influences the total wear of the systems regardless of the load used. Cou-

ple of mechanisms by which the oxides can be detached as wear debris are proposed. Cracking is one of them, as shown in Fig. 18b, c, and e. Thus, the small thickness and also the brittleness of these layers cause their removal from the coating surface under the load. During sliding, this mechanism operates in sequence or simultaneous with other mechanisms involved. The spalling of some layer fragments exposes the coating which is prone to further wear by other wear mechanisms. Harder debris, removed either from the coating or from the ceramic ball will also be present in the contact area, and thus promote another mechanism for the detachment of the oxide layers. This is the rolling and sliding of the debris particles between the sliding surfaces, which once again led to cracking and spalling of the oxides. Once the oxide layer is removed, the amount of system material loss and also the increase in friction depend on the rapidity of the formation of new layer.

The three body abrasive mechanism was seen to follow all the processes described above. Two mechanisms which operate simultaneously are proposed. Thus, when the third body is carbide grains and/or ball fragments, either steel or ceramic, there is a gradual extrusion of the coating matrix. Plastic shearing takes place leading to the removal of binder. As a result, the carbide grains protrude from the coating surface as shown in Fig. 18b and d. They are unprotected and removed during further sliding aggravating the wear process. This is due to the fact that their removal forms additional hard debris which contributes to three body wear mechanism, and also removes ball material especially when steel is used as the counter body. Moreover, carbide removal weakens these locations making further grain removal easier. Hence, successive removal of the grains from these locations creates relatively large pits in the coating, which will initiate further wear. Following this mechanism, the hardening of the matrix and also the increase in the bonding between carbides and the matrix which take place, as seen earlier, during the HIPing post-treatment is influential on the extent of carbide pull-outs and, considering three body abrasive wear, on the degree of the ball material loss.

When the role of the abrasives is played by hard debris detached from the ceramic ball, the wear is more severe. The ceramic debris causes cracking of the oxide layers where they exist and plastic deformation in both carbide grains and binder producing the typical surface topography which consist in parallel grooves running in the sliding direction. This was recognized on all wear tracks except those produced on coatings HIPed at 1200 °C (Fig. 17). The role of this mechanism on the coating material loss can be appreciated from the wear of the as-sprayed coatings which had lower fracture toughness and hardness and they loose material by both the plastic deformation (perhaps microcutting rather than ploughing) and brittle fracture. This aspect of the wear scar of the as-sprayed coating, as depicted in Fig. 17a, shows that the plastic deformation of the coating also took place during abrasive wear. Thus, contrary to the HIPed coatings, abrasive grooves are seen throughout the captured section of the wear

scar of the as-sprayed coating. The debris originated from this process continued this mechanism until the end of the test, and hence resulted in more wear of the as-sprayed test couples.

Although the examination of the wear debris collected at the end of wear tests doesn't give indications of its appearance at the formation, some suggestions on the transformations they suffer from the initial to the final state can be proposed. Thus, flake-like debris was found in most of the tests but also agglomerates of smaller particles, as shown in Fig. 19a. Elemental analysis on flakes-like debris produced in different tests suggest that they have oxide layers. They spalled out of the coatings and were involved in three body abrasive wear which cracked and fragmented them until they reached the sizes seen in Fig. 19. On the other hand, during sliding, particles detached from either the coating or the ball adhere to each other until agglomerates of different sizes form. They can move away from the contact area or get entrapped between the sliding pairs, becoming flattened by the plastic deformation. Although not included in this paper, elemental analysis of the debris indicated a combination of coating fragments, bits of oxide layer and ball material.

Apart from the oxidation wear for which a quantification of the oxide layers for each coating surface is difficult, the mechanisms by which the rest of the processes operate explained the volume loss during the wear test. The lower volume loss of HIPed coatings can be well understood in view of the increased hardness and toughness of the coatings. However, with the increased hardness of coatings, one should expect a higher wear of the counterbody, especially with the lower hardness of the steel balls. However, the results of the total volume loss seen in Fig. 16 indicate lower volume loss of the entire couple, the rationale for which is discussed next.

The relatively higher brittleness of the as-sprayed coatings prones them to a microcracking process which leads to coating damage mainly because of delamination/spallation. The hardnening of the binder during HIPing process decreases the extent of matrix extrusion, and the improved bonding between the carbides and the matrix inhibit carbide pullouts. The increased fracture toughness and the hardness of the HIPed coatings reduces the degree of brittle fracture of the coating and plastic deformation, respectively, which takes place during abrasive wear. Less coating damage reduces the amount of carbide debris within the wear tracks and consequently the oxide layers ensure less wear of the sliding surfaces in the HIPed coatings.

Although the examination of the wear scars at the end of the tests did not provide clear evidence of adhesion, it is not excluded that this mechanism aggravates the wear, and increases friction during the tests. The friction is also amplified by microcracking and the mechanisms involved in abrasive wear, namely plastic deformation and brittle fracture which are energy dissipative processes. Balancing this, it is the presence of oxide layers which keep the friction at a low level acting as solid lubricants for the system. Therefore, a combination of these effects gives the evolution of friction coefficients as shown in Figs. 13 and 14. Although there is no clear difference in the friction between the as-sprayed and the HIPed coatings, at least at the beginning at the tests, the HIPed coatings, particularly those HIPed at 1200 °C show slightly lower friction. Generally, the profiles are relatively smooth except those which corresponded to the tests involving steel balls. The fluctuations are seen mostly at the beginning of the tests and are ascribed to the increase in the apparent area of contact. As the contact area become stable, further variation of the friction coefficients is due to the debris entrapped within the wear scar. This is confirmed by the micrographs of the debris produced during coating/steel tests. They show, beside large flake-like debris, the agglomerates of fine particles mentioned earlier, suggesting that the debris spent a longer time within the contact area hence producing variation and influencing the friction coefficients.

The last issue without which this discussion will not be complete is the apparent proportionality of the friction coefficient with the applied load, although only two different loads were applied. Considering the first law of friction [22], one should expect that the coefficient of friction to be independent of the applied load. However, this is not generally true since it has been shown that the so called "Coulomb friction" (the friction which does not depend of the test conditions). is proven only in few tests. This is confirmed by the narrow range of load employed in this investigation, the results showing that the friction coefficient decreases with increasing the load. The frictional force is therefore not proportional to the applied load. It was observed that the increase in the applied load produced an increase in the frictional force nevertheless not enough to maintain the friction coefficient at a constant value, and represents the complex role of oxide layer and wear debris within the contact region.

5. Conclusions

The tribological performance of the as-sprayed and posttreated thermal spray coatings via hot isostatic pressing was evaluated to investigate the effect that the post-treatment temperature has on the coating wear resistance. The results indicate that the wear performace of the WC–NiCrBSi thermally sprayed coatings can be improved by HIPing process. HIPing the coating without encapsulation by appropriate design of functionally graded coatings and HIPing conditions proved successful. Increasing HIPing temperature produces higher coating wear resistance through the following changes which occur in the coating microstructure, mechanical properties, and residual stress state:

- Microstructural investigations show that phase transformations led to the elimination of brittle phases existing in as-sprayed coatings, and gradually produce harder phases as the temperature of the HIPing process increased.
- 2. As a consequence of the above-mentioned changes in the coating phase composition, the hardness of the HIPed

coatings increases with increasing the HIPing temperature. The qualitative examination of the coating fracture toughness confirms the absence of the brittle tungsten carbide secondary phases or metallic tungsten indicating improvement in coating toughness.

- Microstructural investigations show that considerable diffusion throughout the coating and at the interface of coating layers take place, especially at higher temperatures producing higher bonding and consequently higher coating elasticity.
- The development of uniform compressive residual stress was possible after HIPing the coating at a temperature of 1200 °C.

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