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Preparation and Characterization of Cermet MSZ/Ni–Al Coating Deposited by Flame-Spraying Technique

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10MgO–ZrO_2/Ni–Al cermet powders are sprayed by flame-spray technique onto low-carbon steel substrates' type (API 5L) used commonly in oil industrial. The present study is aimed to investigating the influence of thermal-treatment behaviours on the structural, mechanical, and microstructure evolution properties to check the thermal phase stability at high temperatures. The free-standing cermet samples (of 1.85 mm thick) are heat-treated in air at 1000, 1100, 1200, 1300, and 1350°C, for a 2-hours' ageing time. The test properties are characterized by x-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), wear loss, and Vickers hardness. The results show the deposited cermet coating became thicker and have ideal phase stability with the best mechanical attributes, when the heat treatment is at 1300°C for 2 hours of sintering. Above that, at 1350°C, the microstructural surface shows split-up cracks and pores across the layers, which is not reliable for longer thermal stability. The results also show that zirconium oxide (ZrO_2) has a significant change from cubic (f.c.c. ZrO_2), tetragonal (t-ZrO_2) and monoclinic (m-ZrO_2) structures through the various temperature degrees. These results also show that the wear loss value of the cermet coating is so lower, depending strongly on the porosity and hardness values. Finally, we can say that the heat treatment at 1300°C (2 hours) has a typical uniform lamellar structure and high hardness values, which is reliable for longer thermal stability.

Порошки металокераміки 10MgO–ZrO_2/Ni–Al напорошували методом полуменевого розпорошення на підкладинки з низьковуглецевої криці (API 5L), що зазвичай використовується в нафтовій промисловості. Це дослідження було спрямовано на дослідження впливу поведінки під час термічного оброблення на еволюцію структурних, механічних і мікроструктурних властивостей для перевірки термічної фазової стабільности за високих температур. Окремо стоячі зразки металокераміки (товщиною у 1,85 мм) піддавалися термічному обробленню на повітрі за температур у 1000, 1100, 1200, 1300 і 1350°C упродовж 2 годин старіння. Тестові властивості були охарактеризовані за допомогою рентґенівської дифракції, сканувальної електронної мікроскопії, енергодисперсійної спектроскопії, втрати на зношення та твердости за Віккерсом. Результати показали, що нанесене металокерамічне покриття стало товщим і мало ідеальну фазову стабільність із найліпшими механічними властивостями, коли термічне оброблення відбувалося за 1300°C упродовж 2 годин спікання. Вище цього, за 1350°C, мікроструктурна поверхня показує розколоті тріщини та пори вздовж шарів, що не є надійним для тривалої термічної стабільности. Результати також показують, що оксид цирконію (ZrO_2) має значні зміни від кубічної (ГЦК-ZrO_2), тетрагональної (t-ZrO_2) і моноклінної (m-ZrO_2) структур через різних ґрадусів температури. Ці результати також показують, що значення втрат на зношення металокерамічного покриття настільки нижчими, що сильно залежать від значень пористости та твердости. Нарешті, ми можемо сказати, що термічне оброблення за 1300°C (2 години) має типову рівномірну пластинчасту структуру та високі значення твердости, що є надійним для більш тривалої термічної стабільности.

Key words: thermal-ageing treatment, cermet coating, MSZ/Ni–Al system, thermal-spray coating, mechanical properties.

Ключові слова: оброблення термічним старінням, металокерамічне покриття, система MSZ/Ni–Al, термічне напорошення покриття, механічні властивості.

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1. INTRODUCTION

Thermal barrier ceramic coatings are an attractive combination of structural and mechanical properties, including good thermal stability at high temperatures [1, 2]. The usage of thermal ceramic coatings can be useful in a wide range of applications to enhance the service life of hot parts, oxidation resistance, and protection of the consumed parts by adding thick coatings, especially on oil tubes in refineries [3, 4]. Partially stabilized zirconia ceramic (ZrO_2) exhibits sterling electrical and mechanical properties like good wear resistance, high toughness, high melting point, and excellent thermal resistance stability [5, 6]. Several researchers have regarded that self-bonding metals such as Ni, Al, Cr, Co, or alloys mixing with ceramic metal oxides like Al_2O_3, TiO_2, ZrO_2, WC, and SiC have significant exothermic reactions between them, improving the bond strength, which gives a high adhesion force to the deposited coating, generally called generally a cermet composite material [7]. The flame spray coating technique is the most ideal and superior method to deposit thick cermet coatings and can be used for several engineering implementations, such as a high stability operating temperature on turbine blades, oil pipe protection, and thermal insulation [8]. The previously studied effort by using the yttria partially stabilized zirconia system (YSM) with self-bonding (Ni–Co–Al) metals does not encourage succeeding results for high temperature actuation [9, 10].

In this work, ceramic powders consisting of $10MgO–ZrO_2$ mixed with bond ($Ni_{50}–Al_{50}$) metal powders by ball milling process, getting a nanoparticle size of cermet composite of $10MgO–ZrO_2$/Ni–Al which is named the MSZ/Ni–Al composite system, were sprayed by using flame coating technique under optimum parameters to investigate the effect of heat treatment on the phase stability, microstructure, and wear resistance properties.

2. MATERIALS AND TECHNIQUES

In this study (API 5L), low-carbon steel pipes used in the oil industry are preferred as substrates. Pipes were cut as square-shaped coupons (26.5×26.5 mm with a thickness of 3.51 mm) as substrate pieces. The substrate contains the chemical elements listed in Table 1. Substrates were sand blasting with (Al_2O_3) of particle size of 6 µm at a pressure of 5 bar. The 'blistering' is achieved at a 70° angle and a 20 cm distance to make sure it creates a good roughness with the best adhesion force. The substrates were initially cleaned with alcohol, acetone, distilled water, and hot air drying. The flame spraying technique was carried out immediately after the cleaning type (Rototec 80, Castoline, Eutectic Switzerland) was used, as shown in Fig. 1, *a*.

The sample number six was fixed to holes by turning a holder made from steel, as shown in Fig. 1, *b*. The commercial powders used for the fabrication of the cermet coatings, the oxide ceramic ($10MgO–ZrO_2$) powders, supplied by the Sulzer–Metco company, with a 40–50 µm particle size and partially stabilized by MgO, were used as ceramic coating materials. In addition, nickel–aluminium powder of 55-µm average particle size was used as a bonding $Ni_{50}–Al_{50}$ type (Amdry No. 995). Before spray coatings, the initial morphology of both powders was determined, as shown in Fig. 2, *a*, *b*, by scanning electron microscopy (SEM). The raw cermet of composite $10MgO–ZrO_2$/Ni–Al powders, with a purity 99.6%, was ball milling technique using a high-energy planetary (Home Madel) consisting of stainless-steel jars and 40 balls with a diameter of 10 mm. The rotation speed was 137 rpm. The ball-to-powder weight ratio was set at 40:1 gm. The new product sample for 1 hour of milling was 60 nm in size.

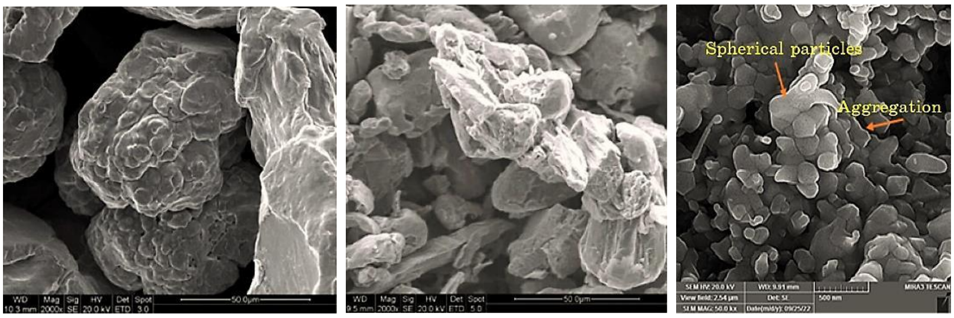
The ideal parameters in the cermet coating process, resulting in

TABLE 1. Chemical elements of oil pipe substrate.

Element	C	Mn	P	S	Cu	Ni	Cr	Mo	V
Weight, %	0.30	1.20	0.05	0.045	0.40	0.40	0.40	0.15	0.08

a *b* *c*

Fig. 1. The flame-spray process during coating: *a*—flame spray device; *b*—turning holder; *c*—sample product.



a *b* *c*

Fig. 2. SEM morphology for initial powders used: *a*—ceramic $MgO–ZrO_2$; *b*—$Ni_{50}–Al_{50}$ alloy bond; *c*—cermet coating of $MgO–ZrO_2$/Ni–Al at RT by flame spring method.

optimized results, are presented in Table 2.

The cermet coating samples were isothermally heat treated in a high-temperature chamber furnace (HTK 20/17, Bremen, Germany) at various temperatures starting from 1000, 1100, 1200, 1300, and 1350°C, respectively, for a 2-hour ageing time. The samples were heated at about 10°C/min to the target temperature and then cooled naturally in the side furnace to room temperature.

TABLE 2. The ideal flame-spray parameters of the cermet coating MSZ/Ni–Al.

No.	Operating parameters	Values
1.	Spray distance	20 cm
2.	Flame-spray temperature	≈ 3000°C
3.	Maximum thickness	1.850 mm
4.	Particle size of $MgO-ZrO_2$	40–50 μm
5.	Particle size of $Ni_{50}-Al_{50}$	55 μm
6.	Oxygen pressure	5–6 bar
7.	Acetylene pressure	2–3 bar
8.	Angle of sand blasting	70°
9.	Rotation number	5
10.	Oxy–Acetylene mixing	3:1

The cermet coating sample was also heat-treated at 1300°C for 6 hours in the furnace to check the influence of time ageing on the thermal phase stability and mechanical properties. The thermal stability was also examined by scanning colorimetry (DSC) (Netszch 404, Germany) with a rate of 5°C min^{-1} up to 1750°C. The structural and phase transformations were carefully studied by x-ray diffraction (XRD) in a Philips diffractometer using philtred CuK_α radiation ($\lambda = 1.540$ Å). The goniometer was set at a scan rate of 0.05°/sec over a 2Θ range. The microstructure and morphology of cermet composite powder particles and as-sprayed coatings at various temperature ranges were investigated using scanning electron microscopy (SEM) equipped with an energy dispersive x-ray analyser (EDAX). The SEM type (Jeol JIB-46 IOF) was used. The microhardness of surface coating values (*HV*) was determined by the average of five tests at a loading of 100 gm during the 15-second period. The microhardness type (Leitz Wetzlar, Germany) was used after the preparation of smoothing and polishing the coating surface. Finally, the wear test method of pin-on-disc sliding was used. The steel rotating disc of 40-*HV* hardness under constant (9 N) load and (1200 cm/min) sliding distances was used as shown in Fig. 3, *a*, *b*. The samples were weighted before and after each test using an electronic balance with an accuracy of 0.002 gm, and losses were recorded.

3. RESULTS AND DISCUSSION

The composition used of both feed stock materials was investigated initially before spray coating, as shown in Fig. 2, *a*, *b*, *c*. It shows the SEM microstructure of $10MgO-ZrO_2$ powder and $Ni_{50}-Al_{50}$ pow-

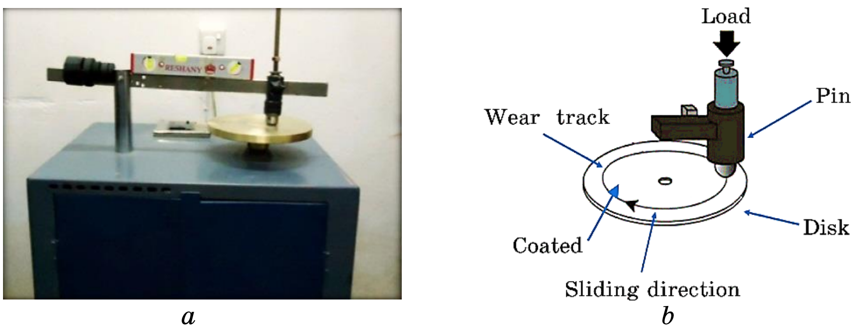


Fig. 3. Wear test of cermet coating MSZ/Ni–Al [11]: *a*—wear pin-on-disk machine; *b*—schematic diagram of the wear test.

der, respectively. Figure 2, *a* shows large agglomerates of spherical particles, shaped in a uniform homogenous condition, while Fig. 2, *b* appears to have sub-angular particles, whose size of Ni_{50}–Al_{50} is of around 50–55 μm, while that of 10MgO–ZrO_2 is of about 40–50 μm. Figure 2, *c* shows the micrograph of the cermet coating after ball milling at 1 hour. As shown, MSZ/Ni–Al particles are spherical-shaped and of uniform size, when the spray distance is of 20 cm.

Figure 3, *a–e* shows SEM micrographs of MSZ/Ni–Al surface cermet coating at different heat-treatment high-temperature values 1000, 1200, 1300, 1350°C, respectively, for 2-hr sintering time. Figure 4, *a* shows the morphology of the cermet surface coating under heat treatment of 1000°C at the sintering time of 2 hr. The surface coating show a high uniform spherical particle distribution of ZrO_2 grains are formed agglomerates with a grain size of around 45–55 μm. The grains of MgO, Ni–Al and ZrO_2 are appears strongly sintered at 1000°C. In addition, the results at heat-treated sample at 1100°C (Fig. 4, *b*) show that the surface coating is similar than as at 1000°C treatment. It shows that most MSZ/Ni–Al particles are also spherical uniformed as agglomeration everywhere on the surface coating, which looks like homogenous condition [12].

When the heat treatments are performed at 1200°C, we have observed that grains are also homogeneously distributed, a good small and large agglomerated microstructure among all grain sizes of MgO, Ni–Al and ZrO_2 particles is shown in Fig. 4, *c*. For further high treatment at 1300°C, as shown in Fig. 4, *d*, it gives the high increment of grain growth significantly different from other treated samples. No any significant of any trace of pores or cracks appeared. It is clear that the steady state for size distribution of particles has been nearly achieved balance more uniform particle size and formed agglomerated strongly as can be seen in Fig. 4, *d*. Above that at 1350°C treatment, spallation, visible pores and cracks

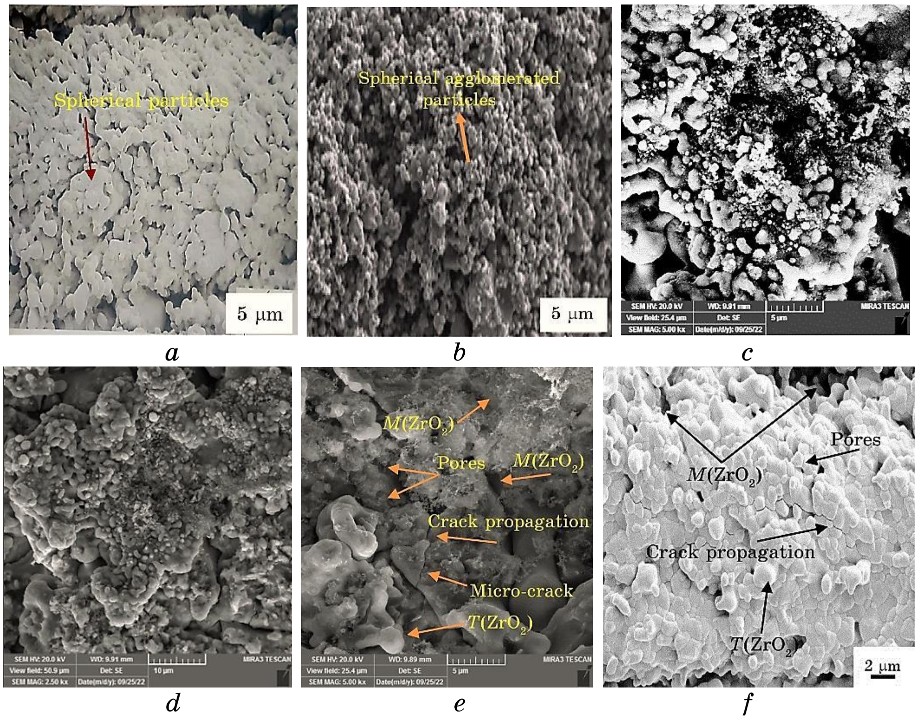
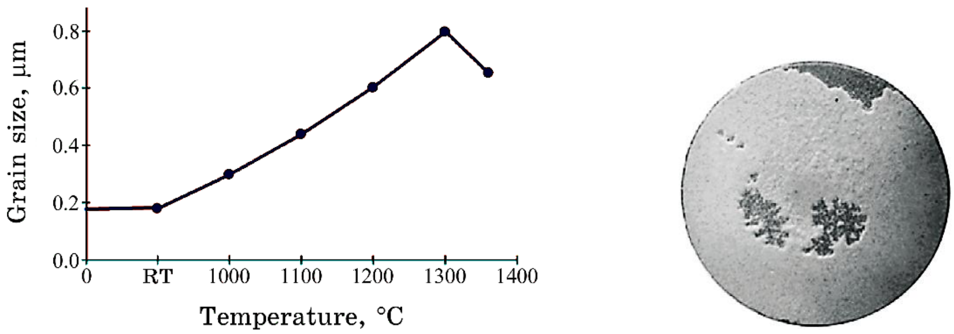


Fig. 4. SEM micrographs of MSZ/Ni–Al surface cermet coatings at different temperature values for 2-hr sintering time. *a*—1000°C; *b*—1100°C; *c*—1200°C; *d*—1300°C; *e*—1350°C, scale 5 μm; *f*—1350°C, scale 2 μm.

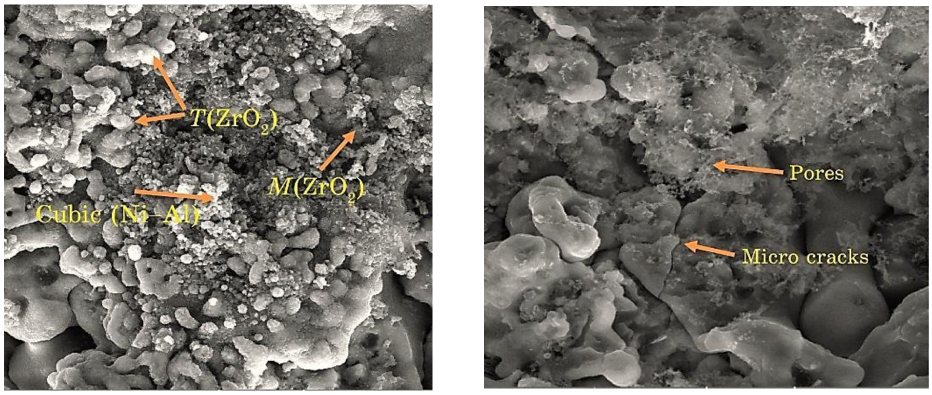
observed at the surface coating, as shown clearly in Fig. 4, *e*. In Figure 4, *f*, SEM morphology structure of the cermet coating MSZ/Ni–Al system at small scale of 2-μm size was taken for more accuracy, which shows the spallation and surface defects clearly [13].

The results also show that the grain size growth started to increase gradually, during the increasing the heat treatment until 1350°C and then dropped suddenly, due to the surface splitting and arising several pores with crack propagation on the top surface cermet coating as shown in Fig. 5, *a*–*b*. These results can be compared between 1000°C and 1300°C that refers obviously the effect of heat treatment on formation high growth rate [14]. In Figure 5, *a* too, we noticed that the grain sizes of cermet coating of MSZ/Ni–Al composite during treated at 1300°C is bigger than, when heated at 1000°C, about more than twice (2.478), which indicates that using treated sample at 1300°C is significantly improved in a uniform microstructure, high adhesion force between layers with high thickness $\leq$ 1.85 mm, which gives a great advantage especially at



a *b*

Fig. 5. *a*—The relationship between the grain sizes and heat-treatment temperatures of surface composite MSZ/Ni–Al coating; *b*—photographs of seceding samples treated at 1350°C for 2-hr sintering time.



a *b*

Fig. 6. SEM micrographs of cermet MSZ/Ni–Al surface coatings after treated at 1300°C as a function of sintering time at: *a*—6 hours; *b*—7 hours.

high thermal-stability applications [14, 15].

Some other attempts have been tested again at a heat-treatment temperature of 1300°C for 6–7 hours to see the effect of sintering time on the microstructure properties of the cermet coating of the MSZ/Ni–Al system, as shown in Fig. 6, *a*, *b*. The results showed that, after 6 hours of sintering, the cermet coating had an atypical structure without any trace of surface defects, as shown in Fig. 6, *a*. For heat treatment for 7 hours, sintering time was used to check the thermal stability of the sample. It appears that degeneration occurs in the surface coating with micropores, which are dominated as result in a long sintering time (7 hr) effect, as shown in Fig. 6, *b*.

Energy dispersive x-ray analysis (EDX) experiments have been conducted on the cermet coating samples at various thermal treatment temperatures of 1000°C and 1350°C, respectively, as shown in Fig. 7, *a*, *b* and Table 3.

It is clearly seen that only the peaks of Zr, Mg, Ni, Al, and O with trace Cr are observed for both samples. Actually, the existence of Cr elements is coming from a production company to increase the adhesion force between the elements of cermet coating [16]. The results also noted that the weight percentages (wt.%) for all elements in both treated samples are very close. This means that the raw materials used in this search are of high pure quality, and the accuracy of the tests is high. In comparison between both samples treated at 1000–1300°C, we found that the samples have agglomerated spherical particle distributions, as shown in Fig. 6. This aggregation may cause a more homogenous structure due to the interaction between the $MgO–ZrO_2$/Ni–Al particles, providing much improved mechanical and structural properties. Indeed, the sample treated at

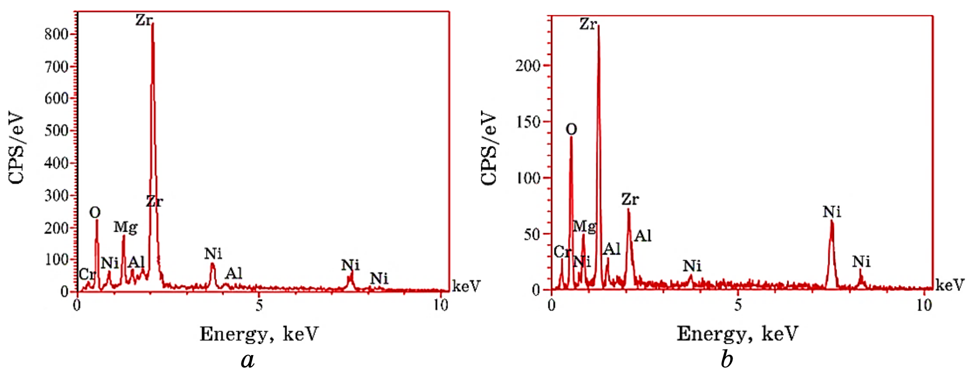


Fig. 7. EDX patterns for cermet coating MSZ/Ni–Al system at various temperatures: *a*—1000°C; *b*—1300°C.

TABLE 3. Analysis of elements by EDX processing at 1000°C and 1350°C for MSZ/Ni–Al coating.

Temperature, °C	Element	Energy level	Weight percentage, %	Total
1000	O	*K*	27.95	99.97%
	Zr	*L*	51.21	
	Mg	*K*	9.67	
	Ni	*K*	5.18	
	Al	*K*	5.96	
1300	O	*K*	29.66	99.51%
	Zr	*L*	55.12	
	Ni	*L*	7.91	
	Al	*L*	6.82	

1300°C for 6 hours of sintering time provided more successfully a strong thermal stability [17].

The structural properties of the cermet coating under different heat-treatment temperatures by XRD RT, 1000, 1100, 1200, and 1300°C for a 2-hour sintering time are investigated as shown in Fig. 8, *a–f*, respectively. Figure 8, *f* shows the sample treated at 1300°C for 6 hours of sintering to see the effect of sintering time on the structural properties of MSZ/Ni–Al coating. Before coating, the cermet powders (RT) show that four phases are indicated, obviously, corresponding to the cubic (f.c.c.-ZrO_2) phase and the tetragonal (t-ZrO_2) phase. The other two phases belong to the cubic (f.c.c.-MgO) and (f.c.c.-self-bonding Ni–Al) phases. All peaks are clearly small in intensity, with broader peaks as shown in Fig. 8, *a*. The powder samples after coating and treatment at 1000°C for 2 hours of sintering time show that the peaks at high 2Θ angles are still wider with small intensity, but, at small angles at $2\Theta \approx 35°$, they show a high intensity of (t-ZrO_2) phase at all the various heat treatments used. The diffraction peaks related to the f.c.c.-ZrO_2 and t-ZrO_2 phases are still dominant during the heat treatment until 1300°C for a 2-hour sintering time, as shown in Fig. 8, *b–f*.

The results also show that phases increase noticeably with increasing heat-treatment temperatures. The peak intensity of the cermet MSZ/Ni–Al coating at $2\Theta \approx 35.30°$ with the t-ZrO_2 phase looks like much stronger intensity than the other phases. Moreover, interest is in studying the effect of sintering time in order to check the thermal stability at high temperatures (1300°C) as a function of the sintering time (6 hr), as shown in Fig. 8, *f*. It shows that a split

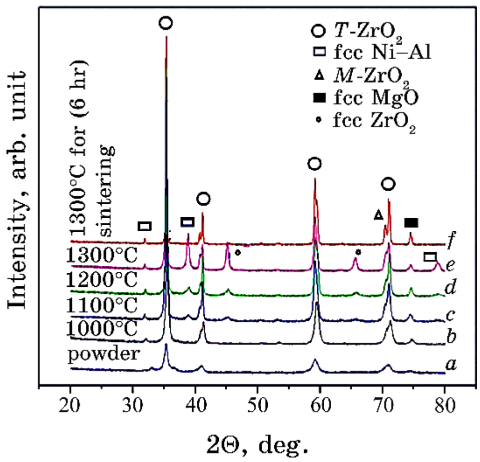


Fig. 8. The XRD structural peaks at various heat treatment of cermet coating MSZ/Ni–Al as functions of treatment temperatures at 2-hr sintering.

peak at a high angle around ($2\Theta \approx 71°$) starts increasing from the initial treatment 1000°C until the severalty state at 1300°C treatment at a sintering time of 6 hours, as shown in Fig. 8, *f*. This splitting phenomenon was observed and accompanied by losing totally the f.c.c.-ZrO_2 phase at the position angles (2Θ) of 45° and 65°, respectively, as shown in Fig. 8, *e*–*f*. The splitting peak actually has two peaks sitting in the same position. As shown by x-ray analysis, it was accompanied by appearing new monoclinic phase *m*-ZrO_2 with *t*-ZrO_2 together, as shown clearly in Fig. 8, *f* [18]. This may be due to the treatment at 1300°C for 6 hours of sintering, which is enough treatment to intensify two (*t*-ZrO_2 and *m*-ZrO_2) phases, giving the best thermal stability [19]. Finally, the x-ray peaks for all measured patterns under several treatments do not show any trace of strange elements, as impurity phases were noticed. This result is actually identical to the results of the EDX test, which also shows no impurities.

Finally, the results of the effective variable heat-treatment temperatures at 2-hr sintering time on the mechanical properties (hardness, grain size, porosity, wear loss) of the cermet 10MgO–ZrO_2/Ni–Al (MSZ/Ni–Al) coating are shown in Table 4. The results of microhardness (*HV*) clearly show that there is no big change, when varying the heat treatment from 1000°C to 1300°C at a 2-hr sintering time, probably, due to the fact that all particles of the composite cermet coating are formed and totally melted in a homogeneous structure [12, 19]. The results also observed at the same time (Table 4) showed a small change in the magnitude of thickness and porosity percentage values during the treatment processes, which were around 1.854-mm thickness and 7.480% porosity, when treated with the cermet the sample at 1300°C for 2 hours of sintering. This means that a thick cermet coating can improve the structural and mechanical properties of the low-carbon steel substrate of oil pipes used in this search, when exposed to an erosive environment at 1300°C, that is proved as most suitable to give the best thermal stability and is ideal for longer temperature life [18, 20].

The microhardness actually depends directly on the porosity percentage, which was found to be about 7.48% after heat treatment

TABLE 4. The wear loss with a sliding distance at 9-N normal load.

Temperature, °C	Porosity, %	Microhardness, *HV*	Grain size, μm
1000	9.86	46.88	0.322
1100	8.89	46.98	0.412
1200	8.04	47.46	0.605
1300	7.48	47.85	0.798
1350	13.77	25.27	0.491

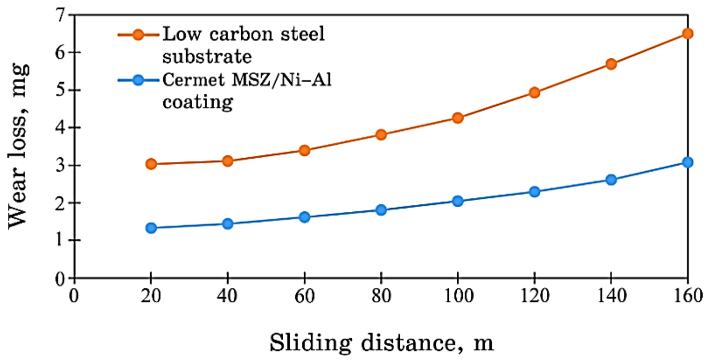


Fig. 9. The wear loss relation to the sliding distance at 9-N normal load effect.

at 1300°C sintering. This may be due to the presence of oxides related to MgO, ZrO_2, NiO, and Al_2O_3 oxides between the coating layers [21]. As the heat temperature continued to increase to 1350°C, a significant reduction in the hardness value was noticed. We believed that the increased cracks, porosity, and other surface defects led to a reduction in the hardness value, as shown in the SEM results (Fig. 4), which show many defects with porosity [22]. The results also clearly show the influence of the heat treatment on the grain sizes of MSZ and Ni–Al samples, as shown in Table 4. It is obviously noticed that grain-size growth started to increase during the increasing thermal treatment until 1300°C, which refers to the effect of high treatment on the formation of high growth. This may be related to the reduction in porosity values accompanied by increases and improvements in structural, microstructural, and mechanical properties [23]. The wear loss amount is related to the sliding distance at 9-N load effect on the oil pipe substrate, low-carbon steel, and into the cermet coating 10MgO–ZrO_2/Ni–Al sample, as shown in Fig. 9. The results show that the wear loss value of the simple cermet coating is lower on the substrate; probably, this is because the hardness value of the low-carbon steel substrate is very lower than that of the cermet coating sample. In our wear loss results for the cermet coating, we found a very strong wear loss depending on the porosity percentage and hardness values. The highest hardness value of 47.85 *HV* with the lowest porosity around 7.48%, having the lowest wear loss roughly of ($3\cdot10^{-3}$ mg) at the 9-N normal load, was observed as shown in Fig. 9.

4. CONCLUSIONS

Cermet mixtures of ceramic 10MgO–ZrO_2 oxide powder with bond metal $Ni_{50}Al_{50}$ alloy powder were deposited by using the flam spray technique. The cermet samples $10MgO–ZrO_2 + Ni–Al$ coating were subjected to heat treatment sintering for 2 hours at various temperatures 1000, 1100, 1200, 1300, and 1350°C with the objective of studying the influence of high temperature ranges on the structural, microstructural, and mechanical properties. Four phases identified by XRD show that the cermet (MSZ/Ni–Al) powders before coating (RT) contain four phases: f.c.c.-MgO, f.c.c.-Ni–Al, f.c.c.-ZrO_2 and the tetragonal phase t-ZrO_2 during different initial temperatures at a 2-hour sintering time. The thermal stability at high temperatures 1300°C was checked by increasing the sintering time to 6 hours. The splitting peak at high angle was observed with the f.c.c.-ZrO_2 phase losing totally and a new monoclinic (m-ZrO_2) phase sharing with t-ZrO_2 phase giving the best thermal stability. The micrograph (SEM) of MSZ/Ni–Al surface coating shows high uniform spherical particle distributions that form agglomerate until 1300°C. Above that, at 1350°C, treatment, spallation, pores, and cracks were clearly observed. The effect of variable heat treatment on mechanical properties has also been observed, with no significant change in microhardness when the heat treatment temperature is varied up to 1300°C. In addition, at 1350°C treatment, a sudden reduction in hardness value to 25.27 HV. The thickness and porosity values during the heat treatment at 1300°C are of 1.854 mm and 7.480%, respectively. In addition, we noticed the highest hardness value of 47.85 HV with the lowest wear loss of roughly ($3 \cdot 10^{-3}$ mg) at 9-N normal load. Finally, this result means that the cermet coating (MSZ/Ni–Al) composite system at 1300°C for 6 hours of sintering can improve its structural and mechanical properties to protect the low-carbon steel of oil pipes at higher temperatures.

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