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Laser surface remelting of plasma-sprayed nanostructured Al_2O_3–13 wt%TiO_2 coatings on magnesium alloy

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ABSTRACT

Plasma-sprayed nanostructured coatings were successfully deposited on an AZ91D magnesium alloy substrate using the as-prepared nanostructured Al_2O_3–13 wt%TiO_2 feedstock and were subsequently remelted by a CO_2 laser. The effects of laser remelting on the microstructure, phase composition and mechanical properties of the ceramic coatings were investigated by scanning electron microscope, X-ray diffractometer and Vickers microhardness tester. The results indicate that the laser remelted coatings exhibit excellent metallurgical bonding to the substrate. Pores and lamellae structures in the as-sprayed coatings have been effectively eliminated and a more compact and homogenous microstructure is achieved after laser remelting. The metastable γ-Al_2O_3 phase in the as-sprayed coatings was transformed to stable α-Al_2O_3 during laser remelting. The microhardness of the remelted coatings was enhanced to 1000–1500 $HV_{0.3}$, which is about 15 times higher than that of the substrate. In addition, with the decrease of laser scanning speeds, the microhardness was increased correspondingly.

1. Introduction

Magnesium is the eighth most abundant element on the Earth making up approximately 1.93% by mass of the Earth's crust and 0.13% by mass of the oceans [1]. As an extremely light metal, magnesium and its alloys have already been extensively used in aerospace, aircraft, automotive, electronics and other fields owing to their excellent specific strength and sound damping capabilities, good hot formability, machinability, and electromagnetic interference shielding, as well as high recycling potential [2]. However, the wider application of magnesium and its alloys is considerably restricted by their poor surface properties such as low hardness, poor corrosion resistance and low wear resistance [3]. Therefore, the surface characteristics of the magnesium alloys need to be improved.

Nanostructured ceramic materials generally possess superior physical, chemical, mechanical, and wear and corrosion properties owing to their quantum size and surface effects [4,5]. Owing to the advantages of simplicity and flexibility, plasma spraying is one of the most appropriate techniques for the fabrication of nanostructured ceramic coatings to provide improved strength, surface hardness, as well as corrosion and wear resistance [6,7]. Al_2O_3–TiO_2 ceramic coatings have been applied as surface coatings in many fields. For the Al_2O_3–TiO_2 system, 3 wt%TiO_2, 13 wt%TiO_2, 40 wt%TiO_2 and 50 wt%TiO_2 have been reported. Among them, the Al_2O_3–13 wt%TiO_2 composition exhibits high toughness, attractive wear and corrosion resistance, and has been most widely used. In addition, a new plasma-sprayed Al_2O_3–13 wt%TiO_2 coating modified by rare earth and derived from nanocrystalline powders has also been reported. The modified coating shows novel and excellent properties, such as bond strength, toughness, abrasive wear and corrosion resistance, compared with their conventional counterparts [8–11]. However, there still remain two major problems concerning plasma spraying. One is the poor coating–substrate adherence, which causes the sprayed materials to peel off under high bending stress or heavy load [12]. The other is the presence of lamellar structures and the microdefects including pores and micro-cracks, which tend to reduce the hardness and deteriorate the wear and corrosion performances of the coatings [13].

Post laser treatment is a promising way to eliminate such defects. Many investigations of post laser treatment have been done on the plasma-sprayed coatings, which may contribute to the elimination of porosity, enhancement of the coating strength and chemical homogeneity, as well as the development of a metallurgical bonding at the coating–substrate interface producing strengthened coating adhesion [14,15]. The conventional Al_2O_3–TiO_2 ceramic coatings on carbon steel and aluminum alloys prepared by plasma spraying and laser remelting have been investigated [16–19]. Nevertheless, few studies have been conducted

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on laser remelting of nanostructured Al_2O_3–TiO_2 coatings. In our previous work, nanostructured Al_2O_3–13 wt%TiO_2 coatings have been prepared on a titanium alloy by plasma spraying and laser remelting [20]. To date, owing to relatively lower melting point and laser absorption coefficient of magnesium alloys, the preparation of such remelted coatings on the magnesium alloys has been rarely reported.

In this study, nanostructured Al_2O_3–13 wt%TiO_2 coatings were deposited by plasma spraying on a magnesium alloy (AZ91D). The as-sprayed coatings were subsequently remelted by a CO_2 laser. The aim of this study is to investigate the effects of laser remelting on the microstructure, phase composition, and mechanical properties of the as-sprayed nanostructured coatings fabricated on the magnesium alloy.

2. Experimental

2.1. Substrate material

The substrate material used for the present study was AZ91D magnesium alloy. The chemical composition (wt%) of the as-received substrate material is listed as follows: 8.5–9.5 Al, 0.90–0.95 Zn, 0.17–0.40 Mn, 0.03 Si, 0.002 Fe, 0.001 Cu, 0.001 Ni, and the balance is Mg. Samples were cut into slabs with the size of 60 mm × 15 mm × 5 mm by wire electrical discharge machining.

2.2. Feedstock reconstitution and plasma spraying

In this study, the feedstock used for plasma spraying was reconstituted agglomerates derived from nanoparticles. The raw material powders consisted of Al_2O_3 (δ and γ phases, 99.9% purity, Degussa Co., Ltd., Germany) with particle size of 20–45 nm, TiO_2 (anatase, 99.9% purity, Nanjing High Technology Nano Materials Co., Ltd., China) with particle size of 20–50 nm, ZrO_2 (tetragonal phase, 99.9% purity, Cug–Nano Materials Manufacturing Co., Ltd., China) with particle size of 20–50 nm and CeO_2 (cubic phase, 99.9% purity, Rare Chem. Co., Ltd., China) with particle size of 20–40 nm. ZrO_2 and CeO_2 were used as additives and their weight contents were about 5%, respectively. All the nanoparticles were mixed by wet ball milling. The time used for wet ball milling was 24 h. An emulsion of polyvinyl alcohol (PVA) was added into the slurry as a binder addition. Then, the slurry was spray dried. Finally, the agglomerated powders were subjected to sintering and plasma treatment. The sintering was conducted in a furnace with a temperature of 1200 °C and sintering period of 3 h. During the plasma treatment, the powders passed a plasma flame generated by a Metco 9MB gun (Sulzer Metco, USA) in a very short time. The SEM micrographs of the as-prepared Al_2O_3–13 wt%TiO_2 feedstock is shown in Fig. 1. The feedstock powders are 20–50 μm in diameter and possess regular spherical morphologies with smooth surfaces.

Atmospheric plasma spraying was applied to deposit the nanostructured coatings on ultrasonic-cleaned and freshly grit-blasted AZ91D magnesium alloy substrates. The surface roughness of the substrates was about 10 μm. A Metco 9 M plasma spray control system with a Metco 9MB gun (Sulzer Metco, USA) was employed. A mixture of Ar and H_2 was used for the plasma gas and Ar was used for the powder carrier gas. The plasma deposition parameters were as follows: (a) current was 600 A, (b) voltage was 65 V, (c) primary Ar gas pressure was about 690 kPa, (d) secondary H_2 gas pressure was about 380 kPa, (e) Ar gas flow rate was 2000 l/h, (f) powder feed rate was 1000–1500 g/h, and (g) spray distance was about 100 mm. The coatings were deposited with a thickness ranging from 300 to 400 μm.

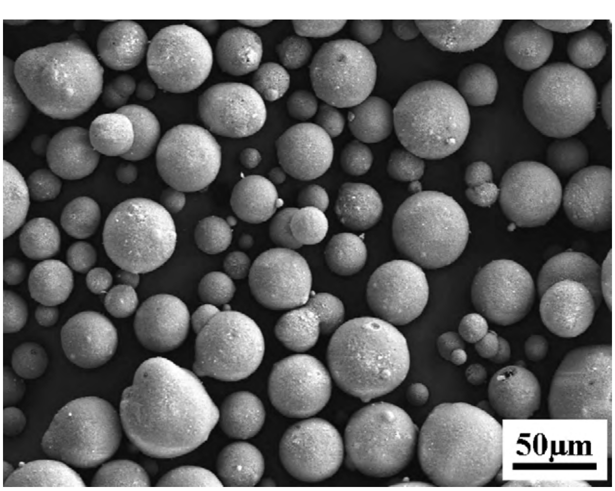

Fig. 1. SEM micrographs of the as-prepared Al_2O_3–13 wt%TiO_2 feedstock.

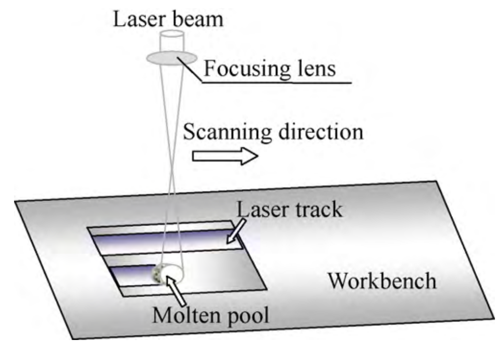

Fig. 2. Schematic illustration of laser remelting process.

2.3. Post laser treatment

The as-sprayed coatings were remelted using a continuous wave CO_2 laser (DL-HL-T5000, China) operating at a wavelength of 10.6 μm with a maximum output power of 5 kW. The laser beam was defocused to a spot of 3 mm in diameter at the surface of the coatings. The laser output power used in this study ranged from 400 to 1200 W, and the scanning speeds of the laser beam ranged from 600 to 1400 mm/min. Argon gas was blown into the molten pool to provide shielding during laser remelting. The details of laser remelting are illustrated in Fig. 2.

2.4. Characterization

The microstructure and phase composition of the feedstock powders and the coatings before and after laser remelting were examined by a scanning electron microscope (SEM, Quanta 200, FEI, USA) and an X-ray diffractometer (XRD, D/max-γB, Japan, with CuKα radiation (λ = 0.15418 nm)). Microhardness of the cross-section of as-sprayed coatings and laser remelted coatings (LRmC) was measured on a Vickers microhardness tester (HV-1000) under 300 g load and 15 s dwell time.

3. Results and discussion

3.1. Microstructure of the coatings

The microstructure of the Al_2O_3–13 wt%TiO_2 ceramic coatings before and after laser remelting was investigated by scanning electron microscopy. The cross-sectional micrographs of the as-sprayed coating and the laser remelted coatings are shown in Fig. 3. The SEM observation reveals that the as-sprayed coating had a typical mechanical bonding between the coating–substrate interfaces. The bonding strength of plasma-sprayed Al_2O_3–13 wt%TiO_2 ceramic coatings to the bond coat was reported to be around 15–30 MPa [21–23]. The coatings with such a low bonding strength can only satisfy the operating requirements under normal service conditions, e.g., relatively low bending stress and environmental temperature. Owing to the relative weak bonding strength, the as-sprayed coatings are subjected to failure when exposed to harsh environments such as large bending stress, high temperature, or severe fatigue cycle. After laser remelting, the remelted coatings possess an excellent metallurgical bonding to the magnesium alloy substrate without interfacial porosity, as shown in Fig. 3b. Metallurgical bonding is produced by chemical bonding between the coating and substrate in intimate contact or even by diffusional interaction. Such metallurgical bonding state with high bonding strength is highly desirable to avoid failure when operated at harsh environments, thus expanding the scope of applications for the coatings.

Fig. 4 shows the surface micrographs of the ceramic coatings before and after laser remelting, which suggests distinct microstructure features at the surface layer of the coatings. Because the coatings were deposited by plasma spraying technique, the

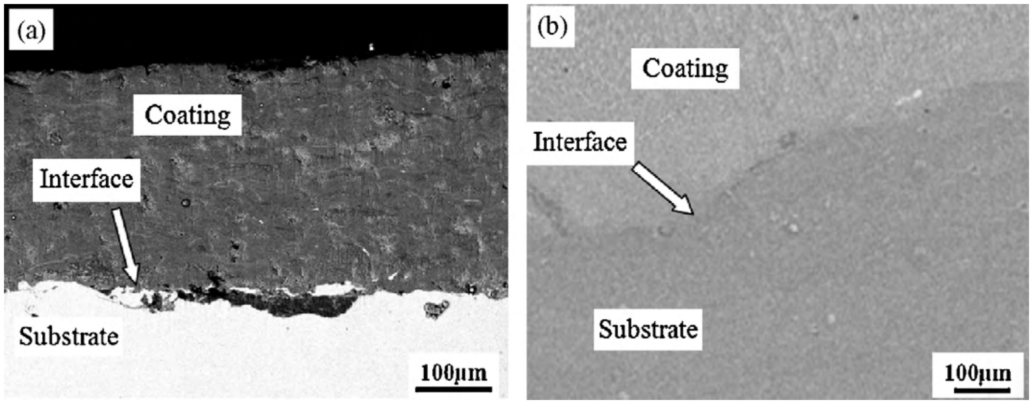

Fig. 3. Cross-sectional micrographs of (a) the as-sprayed coating showing a mechanical bonding between the coating–substrate interface, and (b) the LRmC showing an excellent metallurgical bonding of the coating–substrate interface.

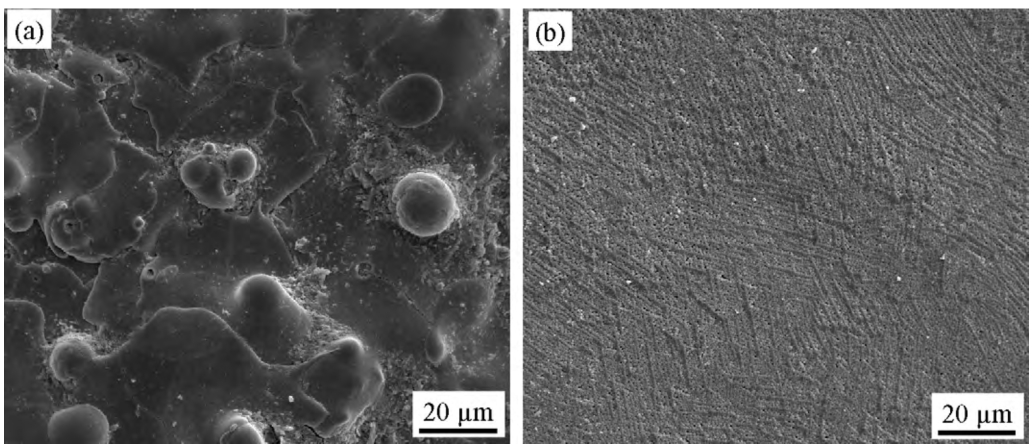

Fig. 4. Surface micrographs of (a) the as-sprayed coating, and (b) the LRmC.

surface microstructure of the as-sprayed coatings reveals the characteristics of porosity and irregular structure of lamellar splats. After laser remelting, the LRmC possess compact and relatively smooth surface microstructure. The SEM observation has demonstrated that the surface microstructure of the as-sprayed ceramic coatings is considerably refined by the laser remelting treatment. The microstructure homogeneity is improved and the pores and lamellae structures are eliminated, which is beneficial to the enhancement of microhardness. It is noted that the additives (ZrO_2 and CeO_2) may also lead to the improvement of compactness, microstructure homogeneity and mechanical properties of the ceramic coatings [8].

The as-sprayed Al_2O_3–13 wt%TiO_2 coatings exhibit a unique bimodal microstructure, which has been widely discussed in the early investigations [23–25]. The as-sprayed coatings are composed of two distinct regions, a fully melted region and a partially melted region, as shown in Fig. 5a and b, respectively. The fully melted region consists of lamellar splats derived from fully melted feedstock, while the partially melted region consists of particulates of three-dimensional net structure derived from partially melted

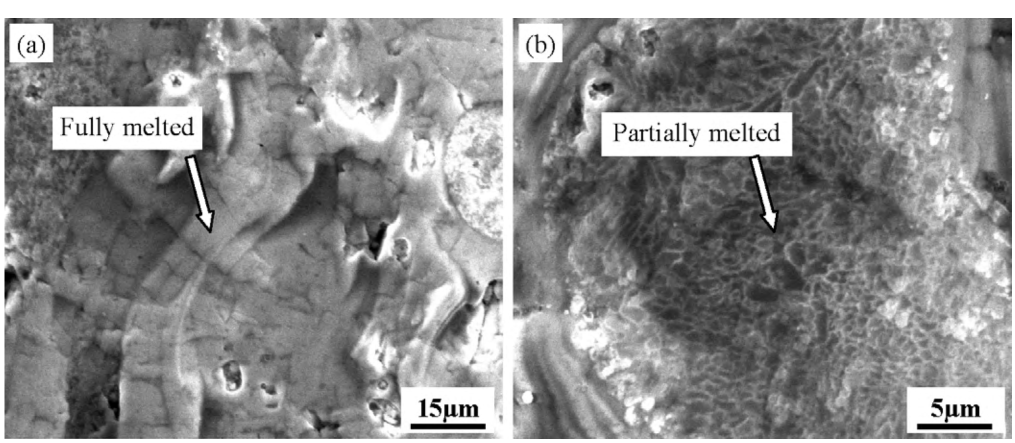

Fig. 5. Cross-sectional micrographs of the as-sprayed coating showing the bimodal microstructure: (a) fused structure derived from fully melted feedstock, and (b) the three-dimensional net structure derived from partially melted feedstock.

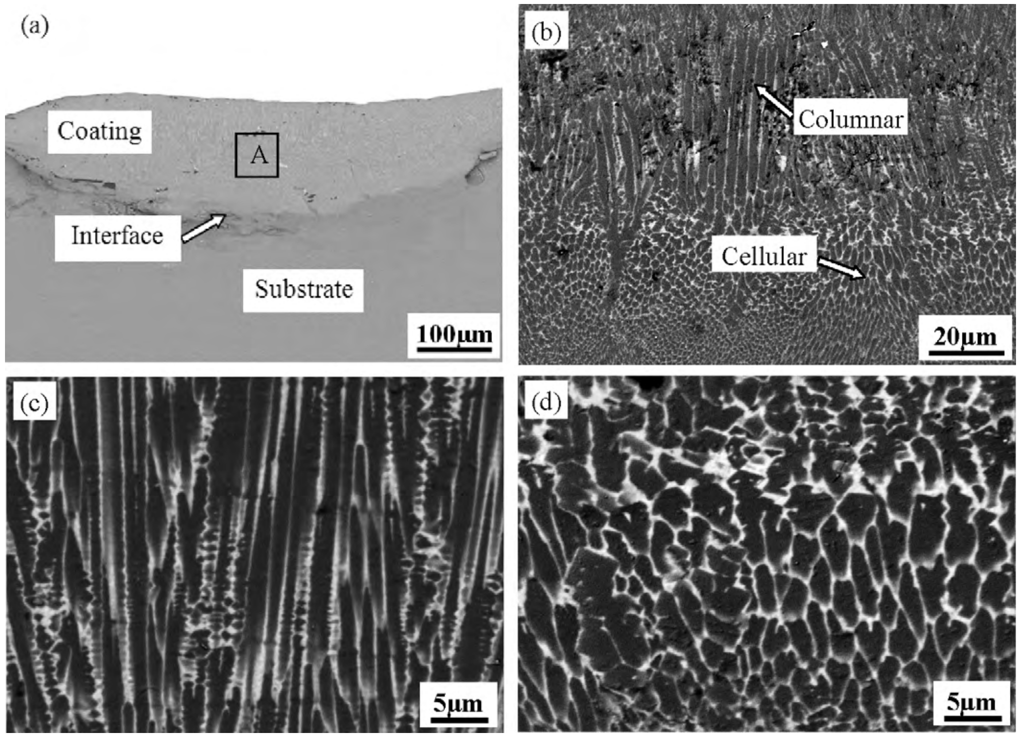

Fig. 6. Cross-sectional micrographs of the LRmC: (a) a typical molten pool, (b) micrographs showing the mixed microstructure features of the columnar structure and cellular structure, corresponding to region A in (a), (c) detailed microstructure features of the columnar structure, and (d) detailed microstructure features of the cellular structure.

feedstock. It is noteworthy that pores and micro-cracks can be occasionally observed in the as-sprayed coatings.

During laser remelting, the as-sprayed coatings and the top substrate were heated to a molten state by the high-energy laser beam to form a molten pool. Fig. 6a shows a typical molten pool of the LRmC. It proves that compact microstructure has been obtained after laser remelting. The defects such as pre-existing pores and micro-cracks of the as-sprayed coatings have been eliminated. In the LRmC, the lamination structure of the as-sprayed coatings has been replaced by a much denser structure. Higher magnification micrographs reveal that the LRmC consisted of columnar structure and cellular structure, as shown in Fig. 6b. More detailed microstructure features of the columnar structure and cellular structure are shown in Fig. 6c and d, respectively. The columnar structure has been found to approximately grow along the direction of the heat flow (vertical to the surface of the coating). This microstructure features are close to those reported by Gao et al. [26] in their study of laser-cladding Al_2O_3 ceramic coating on AZ91HP magnesium alloy. During the laser remelting process, the temperature of the coating surface could reach up to 3000 °C [27], the as-sprayed coating and the top substrate melt simultaneously and crystal refreezing would occur, then some columnar crystal grows along the direction of heat flow. On the other hand, owing to the Gaussian distribution of the laser energy along the diameter of the laser spot, the boundary areas of the laser molten pool were less heated. Further, rapid solidification process restricts the diffusion at these regions, so that a cellular structure can be formed. The cellular structure looks like the three-dimensional net structure found in the partially melted regions of the as-sprayed coatings.

It is noteworthy that the compactness of the coatings has been dramatically improved by laser remelting. The lamellar defects, pre-existing pores and cracks of the as-sprayed coatings have been eliminated. Laser remelting is a process of rapid heating and cooling cycle. Both the rapid remelting and solidification process lead to structural refinement and compactification of the LRmC. The possible presence of micro-cracks in the surface and cross-section of LRmC has been extensively discussed by researchers in their works [28,29]. In the present study, the surface and cross-section of the ceramic coatings remelted at optimized parameters are generally free of micro-cracks.

3.2. Phase composition

Fig. 7 shows the X-ray diffraction patterns of the as-prepared Al_2O_3–13 wt%TiO_2 feedstock, the as-spayed coating and the LRmC. It is evident that the as-prepared Al_2O_3–13 wt%TiO_2 feedstock is composed of α-Al_2O_3, γ-Al_2O_3, Brookite-TiO_2, Rutile-TiO_2 and ZrO_2. After plasma spraying, α-Al_2O_3, γ-Al_2O_3 and Rutile-TiO_2 are

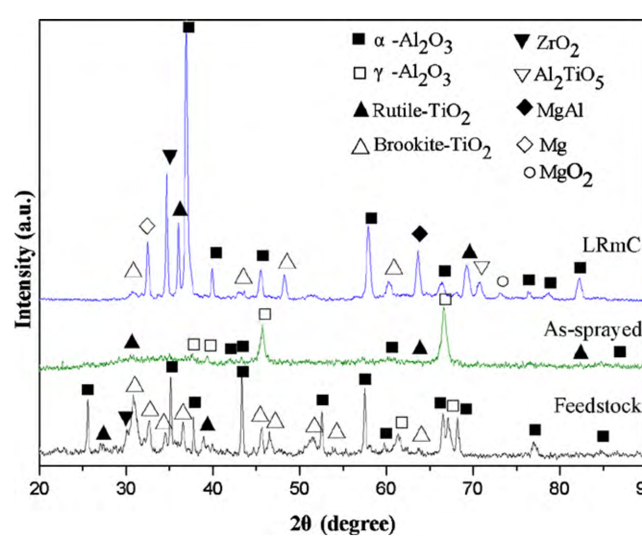

Fig. 7. XRD patterns of the as-prepared Al_2O_3–13 wt%TiO_2 feedstock powder and coatings before and after laser remelting.

present in the as-sprayed Al_2O_3–13 wt%TiO_2 coating. After subsequent laser remelting, α-Al_2O_3, Rutile-TiO_2, Brookite-TiO_2, ZrO_2, Al_2TiO_5, MgAl, Mg and MgO_2 are found in the LRmC.

During plasma spraying, the formation of metastable γ-Al_2O_3 phase was attributable to its lower nucleating free energy and the rapid solidification process. In the LRmC, the metastable γ-Al_2O_3 phase was transformed to stable α-Al_2O_3, which possesses superior mechanical properties than γ-Al_2O_3. In the molten pool remelted by the high-energy laser beam, TiO_2 tended to react with Al_2O_3 during the remelting process and Al_2TiO_5 could be formed at high temperatures above 1200 °C, according to the phase diagram of Al_2O_3–TiO_2 system [30]. The high cooling rate of the coating materials after laser remelting led to the formation of Al_2TiO_5 in the LRmC at room temperature. The rapid cooling of the coating materials restricts the transformation of the Al_2TiO_5 phase into the phases compatible with the phase diagram. Similar observations have also been reported by several authors [18,19]. In addition, when exposed to high temperature in the molten pool, part of Mg tended to react with Al owing to their increased reactive activities, and intermetallic MgAl phase was formed.

3.3. Hardness of the coatings

The microhardness of the ceramic coatings before and after laser remelting is presented in Fig. 8. The microhardness of the as-received AZ91D magnesium alloy substrate falls in the range of 60–90 $HV_{0.3}$, while the microhardness of the as-sprayed coating is in the range of 700–1000 $HV_{0.3}$. The preparation of the as-sprayed ceramic coatings is an effective way to provide the magnesium alloy substrate with a hardened surface layer exhibiting considerable hardness.

The ceramic coating has a low thermal diffusivity and low thermal conductivity. The thermal conductivity of Al_2O_3 is lower than 30 W m^{-1} K^{-1} and that of TiO_2 is lower than 10 W m^{-1} K^{-1} [19]. During laser remelting, the surface temperature of the ceramic coating could exceed 3000 K. Owing to the characteristics of rapid heating and solidification of laser remelting, the temperature gradient from the coating surface down to the substrate can be up to 10^6 K/m. After laser remelting, a graded distribution of hardness was found in the LRmC, which corresponds to three regions: the coating zone, the heat affected zone (HAZ) and the substrate. The coating zone is a remelted zone, which was subjected to the remelting and resolidification process, while the HAZ was only subjected to the heating and cooling process with no melting involved [31]. As shown in Fig. 8, the microhardness of the LRmC was further increased to 1000–1500 $HV_{0.3}$, which is much higher than that of the as-sprayed coatings. The microhardness of the as-received magnesium alloy substrate is about 80 $HV_{0.3}$. After laser remelting, the microhardness of the substrate near the coating–substrate interface is slightly increased to about 120 $HV_{0.3}$. It is noted that the microhardness of the LRmC is about 15 times higher than that of the as-received magnesium alloy substrate. With the help of laser surface remelting, the preparation of a composite system of the base magnesium alloy with a protective Al_2O_3–13 wt%TiO_2 surface coating can be an optimum choice in combining a wide range of superior properties, such as excellent specific strength, sound damping capability, good hot formability, machinability, electromagnetic interference shielding, and high surface hardness. The structure compactness as well as high hardness of the LRmC may also be suggested to offer improved wear resistance for the base material.

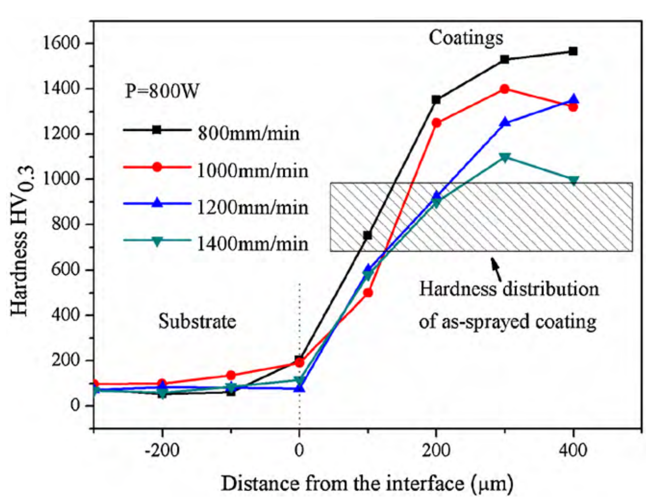

Fig. 8. Hardness of the as-sprayed coating and LRmC remelted at different laser scanning speeds.

In addition, with the decrease of the laser scanning speeds from 1400 to 800 mm/min, the microhardness of the LRmC was increased. Under a constant laser output power, the lower the scanning speed, the more adequate is the melting of the coating materials, and thus a more homogenous and compact structure, which corresponds to higher hardness.

The automotive industry always strives to achieve light-weight components to reduce energy consumption and to meet environmental requirements. The use of magnesium is a solution but this choice requires surface treatments to improve its poor surface characteristics. The preparation of such a hardened LRmC for the protection of the magnesium substrate would be of great importance.

Since this is the initial attempt to explore the laser surface remelting of plasma-sprayed nanostructured Al_2O_3–13 wt%TiO_2 coatings on the magnesium alloy, there are many issues to be addressed and further investigated. As the laser remelting process is a complicated non-equilibrium process involving rapid melting and solidification, the surface tension gradient, temperature distribution and inhomogeneous flow characteristics of the molten pool as well as their effects on the microstructure and properties of the LRmC need to be clarified in future research.

4. Conclusions

The results clearly indicate that laser remelting is suitable for the fabrication of dense Al_2O_3–13 wt%TiO_2 coatings on AZ91D magnesium alloy. It can be concluded that: (1) the as-sprayed coatings exhibit a unique bimodal microstructure consisting of a fully melted region and a partially melted region. The remelted coatings possess an excellent metallurgical bonding to the substrate without interfacial porosity. Pre-existing defects and lamellae structure of the as-sprayed coatings were eliminated after laser remelting and a more compact and homogenous structure was obtained; (2) the XRD analysis proves that the metastable γ-Al_2O_3 phase formed in the as-sprayed coatings was transformed to stable α-Al_2O_3 during laser remelting, and Al_2TiO_5 and MgAl phases were also found in the LRmC; (3) the microhardness of the as-sprayed coating is in the range of 700–1000 $HV_{0.3}$, while the microhardness of the LRmC was enhanced to 1000–1500 $HV_{0.3}$, which is about 15 times higher than that of the magnesium alloy substrate. With decreasing the scanning speed of the laser beam, the microhardness of the LRmC was increased. Laser remelting is a promising means to provide an ideal surface protective layer with high hardness and excellent bonding strength for the magnesium alloy substrates. This further widens their scope of applications, especially in harsh environments with abnormally heavy load and severe wear.

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