Fabrication of *in-situ* **synthesized TiC particles reinforced composite coating by powder feeding laser cladding**

SEN YANG∗

Department of Materials Processing, Graduate School of Engineering, Tohoku University Aoba-yama 02, Sendai 980-8579, Japan; Department of Mechanical Engineering, Tsinghua University, Beijing 100084, People's Republic of China E-mail: syang@material.tohoku.ac.jp

WENJIN LIU, MINLIN ZHONG

Department of Mechanical Engineering, Tsinghua University, Beijing 100084, People's Republic of China

ZHANJIE WANG, HIROYUKI KOKAWA *Department of Materials Processing, Graduate School of Engineering, Tohoku University Aoba-yama 02, Sendai 980-8579, Japan*

Laser cladding is one of the most useful surface engineering technologies for its special advantages and is extensively used in surface modification of metallic materials [1]. A glance at the recent literature on laser surface processing shows increased interest in depositing composite coatings containing various volume fractions of hard particles. The substrate materials included carbon and alloy steels, aluminum alloys, and titanium alloys [2–5]. The coating materials included Fe-base, Ni-base, Co-base alloys with various ceramic powders of WC, TiC, SiC [6–10], etc. In all of ceramic particles, TiC has high hardness, modulus and rather high flexural strength; hence, it is widely used as the reinforced phase of composite materials [11].

Up till now, the majority of reinforced phases are directly added into coating materials. It is unavoidable that crack may propagate from the interface between the reinforcement and the matrix when the surface of particles is not clean or is polluted. In the recent decade, a novel approach, *in-situ* composite method, has been extensively studied to produce composite materials, by which the interface problem could be avoided [12]. Previous work by the present authors has shown that an *in-situ* TiC particulate-reinforced, nickel-based, alloy composite coating could be produced by laser surface remelting a mixture of nickel based alloy, graphite and titanium preplaced on carbon steel, which exhibited very high microhardness [13] (the maximum $HV_{0,2}850$. But there exist some limits during actual application for preplaced coating, such as uniformity of the thickness, coating efficiency, and the shape of the component.

In recent years, co-axial laser cladding technique has gained extensive application in laser surface modification [13] and in rapid tooling production [14], because it is much more flexible and convenient than that of the traditional laser cladding technique, and much easier to be realized automatization. So the present study aims at investigating the feasibility of *in-situ* synthesizing a TiC dispersedly reinforced nickel-based alloy composite layer on AISI1045 steel by powder feeding laser cladding. The microstructure characteristics and the property of coating layer have been studied.

The substrate was AISI1045 steel**,** whose nominal chemical composition in wt.% was: $0.42-0.52$ C, $0.17-$ 0.37 Si, 0.35–0.65 Mn, Ni < 0.25, Cr < 0.25, P < 0.04, $S < 0.04$, balance Fe. The rectangular specimens of $60 \times 15 \times 15$ mm were cut from alloy plate, and the cladded surfaces were ground to a surface finish of $Ra = 0.2$ mm. All specimens were rinsed with ethanol followed by acetone prior to laser cladding. The powder mixtures of the coating alloy were prepared from pure titanium (99.7 wt%, 200 mesh), graphite coated with nickel (180 mesh, 25 wt.%C) and self-fluxing Ni-Cr-B-Si-C powders (200 mesh) in a fixed ratio. The amount of titanium and graphite was 15 wt.%, and the ratio of titanium to graphite powder corresponded to that of stoichiometric TiC. The composition of nickelbased alloy powder is listed in Table I. $GaF₂$ powder was used as flux and was mixed with the other alloy powders at a ratio of 5 wt.%.

Laser cladding was conducted with a PRC-3000 continuous wave $CO₂$ laser with a TH-1 powder feeding system and a coaxial nozzle. The laser output power varied between 1.4 and 1.9 kW, the laser beam scanning velocity was from 3 to 10 mm/s, the powder feeding rate was 3.26 g/min, and the diameter of the laser beam spot was fixed at 3 mm. In order to prevent the melted pool from heavy oxidation, high-purity argon gas was used as shielding gas through the coaxial nozzle at 6 l/min. Fig. 1 is the schematic diagram of the powder feeding laser cladding system.

Phase identification was carried out on a Rigaku D-MAX X-ray diffractometer (XRD) with CuKa radiation operating at 40 kV and 120 mA, and the XRD specimens were cut parallel the scanning direction from the top surface of the coating. Microstructural characterization of the coating was performed with a NEOPHOT32

[∗]Author to whom all correspondence should be addressed.

TABLE I Composition (wt.%) of nickel-based alloy

Figure 1 Schematic diagram of coaxial powder feeding laser cladding system.

optical microscope and JSM-6301F scanning electron microscope equipped with a Link ISIS6566 facility for energy-dispersive spectroscopy (EDS) to characterize the elemental distribution in a semiquantitative manner. Metallographic specimens from the transverse cross-section of the cladding track were carefully prepared using the standard metallographic techniques. The etching agent was a mixture of 10 ml HF and 90 ml HNO₃. Microhardness distribution in the coatings was measured with a load of 200 g by using a HX-200 microsclerometer.

Fig. 2 is a typical micrograph of the macro-shape of the laser cladding coating from transverse cross section. It can be found that there existed an excellent metallurgical bond between the coating and the substrate, and no cracks. However, there existed some pores or holes, which was a general phenomenon during powder feeding laser cladding, and caused by the engulfed gas during powder feeding and unmelted powder; but under

Figure 2 Typical macrograph of the coating layer.

Figure 3 XRD spectrum of the coating layer.

optimal processing parameters, a sound coating can be obtained. Becuase the laser beam was not coaxial with the nozzle, the coating layer was asymmetrical. Also because there always existed some unmelted powder, which was pasted on the surface of the coating layer, the surface of coating layer was rough. However, the surface quality could be modified after laser surface remelting.

Fig. 3 shows the XRD spectrum of the coating layer. Owing to the interplanar distances corresponding to diffraction peaks of possible phases in the coating were close to each other, and non-equilibrium effects of the rapid remelting and solidification in laser cladding resulted in an extension of saturation and distortion of the lattices, it was difficult to identify the phases present in the coating. However, by comparing and analyzing, it can be found that the cladding coating was mainly composed of γ -Ni, TiC, CrB, M₂₃C₆ and TiB₂, which means that the TiC particles can be formed *in-situ*.

Fig. 4 shows the representative microstructures from transverse section of the coating layer at a scanning velocity of 3 mm/s, 1.5 kW output power, and 3.26 g/min powder feeding rate. Owing to the planar growth in the bottom of the molten pool [16], there existed a white band between coating and substrate (see Fig. 4a), which ensured the excellent metallurgical bond between the coating and the substrate. With the increase of distance from the bottom of the coating, the solidification rate rapidly increased and the temperature gradient rapidly decreased, which led the microstructure to change into cellular/dendrite (Fig. 4a, b). The microstructure of the coating layer was composed of γ -Ni dendrite, interdendritic eutectic (γ -Ni and M₂₃C₆), TiC particles, and a small amount of CrB and $TiB₂$. Because the coating was produced by multi-layer laser cladding (three layers), the microstructure was repeatedly heated; there existed a large quantity of *in-situ* formed TiC particles in the bottom of the coating, while only a small amount of TiC particles was present in the bottom of the coatings when using preplaced coating technology [13]. TiC particles with a diameter of about 2 μ m were dispersed in the γ -Ni matrix or interdendritic regions. In the middle of the coating, TiC showed a flower-like shape (see Fig. 4c), which was due to the *in-situ* reaction between graphite and titanium not being finished

Figure 4 Microstructure of the laser cladding layer. (a) Bottom, (b) local magnification of (a), (c) middle, (d) top of cladding layer.

completely. However, there was no longer un-reacted graphite coated with nickel in the upper section of the coating when the coating was remelted once again using the same processing parameters without powder feeding. TiC particles became much larger, and the maximum size of TiC particles was more than $7 \mu m$ (see Fig. 4d). The constituents of the particles in Fig. 4 are shown in Table II, which further confirms that the particles are mainly composed of Ti and C, and the ratio of Ti and C element is nearly 1.

Fig. 5 shows a typical microhardness profile of the coating. Microhardness within the coating gradually decreased from the top surface to the bottom; the maximum value could reach $HV_{0.2}$ 1100, which was 4.5 times larger than that of the substrate. It is noted that there was no sudden transition from the coating to the substrate in the hardness, which indicated an absence of a sharp demarcation in material properties across the

TABLE II Composition of the particles in Fig. 4

Elements	At. $%$	$Wt.\%$
C	40.16	14.35
Ti	55.68	79.32
Si	0.37	0.31
Cr	2.91	4.50
Fe	0.06	0.10
Ni	0.82	1.43
Total	100.00	100.00

Figure 5 Microhardness distribution within the coating.

interface. However, there existed a fluctuation of microhardness between layers, the main reason was that there were less TiC particles below every layer.

In conclusion, a nickel-based alloy composite coating reinforced with TiC particles has been successfully prepared on AISI1045 steel by powder feeding laser cladding. The dispersed TiC particles could be introduced by *in-situ* reacting on of titanium and graphite during laser processing, instead of TiC particles being added into the molten pool directly. An excellent metallurgical bond between the coating and the substrate was obtained. The microstructure of the coating was composed of γ -Ni, TiC, CrB, M₂₃C₆ and TiB₂, TiC particles dispersed in γ -Ni dendrite or interdendritic. The microhardness of the coating showed variation in gradient; the maximum value was $HV_{0.2}1100.$

Acknowledgments

One of the authors (S. Yang) is grateful to Japan Society for the Promotion of Science for offering a JSPS fellowship. The authors would like to express their gratitude to 985 Science Foundation of Tsinghua University (Grant No. 081100500), Innovation Foundation of Astronautic Science and Technology, Postdoctoral Science Foundation of China and Postdoctoral Science Foundation of Tsinghua-Zhongda for financial support.

References

- 1. P. J. E. MONSON and W. M. STEEN, *Sur. Eng.* 6 (1990) 185.
- 2. A. H. WANG, C. ^S . XIE and J. H. NIE, *Mater. Sci. Technol*. **15** (1999) 957.
- 3. S. ZHANG, C. H. ZHANG, W. T. WU and M. C. WANG. *Acta Metal Sinica* **37** (2001) 315.
- 4. X. W. W U, X. Y. ZENG, B. D. ZHU, Z. Y. TANG and K. CUI, *Chin. J. Lasers* **24** (1997) 570.
- 5. A. AGARWAL and N. B. DAHOTRE, *Int. J. Refract. Metals Hard Mater*. **17** (1999) 283.
- 6. J. H. ABBOUD and D. R. F . WEST, *J. Mater. Sci. Lett*. **10** (1991) 1149.
- 7. Q. L I, T. C. LEI and W. Z. CHEN, *Surf. Coat. Technol*. **114** (1999) 278.
- 8. J. PRZYBYLOWICZ and J. KUSINSKI, *J. Mater. Proc. Technol*. **109** (2001) 154.
- 9. H. J. NIU and I. T. H. CHANG, *Metallics. Trans. A*. **31** (2000) 2615.
- 10. Q. M. ZHANG, J. J. HE, M. L. ZHONG and W. J. LIU, *Surf. Coat. Technol*. **20** (2003) 140.
- 11. S . C. TJONG and Z. Y. M A, *Mater. Sci. Eng. R* **29** (2000) 49.
- 12. D. LEWIS III, M. SINGH and S . G. FISHMAN, *Adv. Mater. Proc*. **7** (1995) 29.
- 13. S . YANG, M. L. ZHONG and W. J. LIU, *Mater. Sci. Eng. A* **343** (2003) 57.
- 14. R. JENDRZEJEWSKI, A. CONDE, J. DEDAMBORENEA and G. SLIWINSKI, *Mater. Design* **23** (2002) 83.
- 15. G. T. LEWIS and E. SCHLIGENGER, *Mater. Design* **21** (2000) 417.
- 16. J. W. RUTTER and B. CHALMERS , *Can. J. Phys.* **31** (1953) 15.

Received 11 November 2003 and accepted 18 November 2004