Effect of tempering on microstructure and mechanical properties of cast iron rolls laser alloyed with C-B-W-Cr

R. Zhou\textsuperscript{1,3}, G.F. Sun\textsuperscript{2,\ast}, K.K. Chen\textsuperscript{1}, Y.Q. Tong\textsuperscript{3}

\textsuperscript{1} School of Mechanical and Metallurgical Engineering, Jiangsu University of Science and Technology, Zhangjiagang 215600, China
\textsuperscript{2} School of Mechanical Engineering, Southeast University, Nanjing 211189, China
\textsuperscript{3} School of Mechanical Engineering, Jiangsu University, Zhenjiang 210013, China

Abstract: Laser alloyed layer with C-B-W-Cr powders on high-Ni-Cr infinite chilled cast iron roll was tempered at 500\textdegree C for 60 mins and cooled to room temperature in the furnace. Optical scanning microscopy and X-ray diffractometer were used to investigate the microstructure and phases of the layer. A microhardness tester and wear tester were used to test the microhardness distribution, friction and wear behavior. Ferrite was formed in the tempered layer. Metastable carbides changed to stoichiometric carbides after tempering. FWHM (full width at half maximum) of the first primary peak was decreased by tempering. Microhardness decreased by 44\%. Friction coefficient of the tempered layer exhibited gentle increase and a stable value of 0.6 was achieved for both the tempered layer and substrate. Relative wear resistance of the tempered layer was 1.11 times that of the tempered roll substrate. Mechanism of the effect of tempering on microstructure, FWHM, microhardness and wear properties were discussed.

1. Introduction

Rolls are the main deformation tools in rolling mills. They withstand very high loads, thermal fatigue, and severe environmental attack. As a result, they often fail due to cracks, spalling, and wear [1, 2]. High-Ni-Cr infinite chilled cast iron rolls are widely used in manufacturing hot rolled plate, wire rod, bar, et al due to their good wear resistance, viscous resistance, thermal cracking resistance and spalling resistance. The requirement for high wear resistance is improved due to the increased rolling speeds and loads [3]. Strengthening technologies are effective ways to prolong service life of rolls.

Laser surface modification has some unique advantages over other surface modification techniques, such as low heat input to the material, strong metallurgical bond between the composite layer and substrate, the formation of a non-equilibrium or amorphous phase as well as homogenization and refinement of the microstructure, all without affecting the substrate properties [4-7]. Laser surface alloying (LSA) has improved the wear resistance of different rolls to 1.6-8.8 times [2]. Ceramic materials are often added to metal matrix to form composite coatings to improve the wear resistance of the matrix [8,9].

In view of the preceding information, the authors have investigated the microstructure and microhardness of the high-Ni-Cr infinite chilled cast iron rolls laser alloyed with C-B-W-Cr composite powders [10]. Results indicated that there were lots of cracks in the alloyed layers. Microhardness can be improved to an average value of 1001HV\textsubscript{0.05}, which is 1.53 times that of the roll substrate. This
paper aims to investigate the effect of tempering on microstructure, phases, FWHM (full width at half maximum) of presented peaks, microhardness and wear behavior of the alloyed layers on high-Ni-Cr infinite chilled cast iron rolls.

2. Experimental details

2.1 Sample preparation

Fabrication of the alloyed layer was from the reference [10]. Sample C was fabricated with the laser power of 7.2 kW, spot diameter of 1.5 mm, scanning speed of 11 m/min, and an overlap ratio of 33.3% was chosen to be tempered. Tempering of the alloyed layer was processed in a furnace with a ramping rate of 20°C/min, a holding temperature of 500°C and a holding time of 60 min, followed by a furnace cooling to the room temperature in about 20 h.

The tempered sample was sectioned, mounted, grounded polished, and etched with an etchant containing 20 g FeCl₃, 50 mL HCl and 100 mL H₂O to reveal the microstructure by immersion technique.

2.2 Morphology and phase analysis

The morphology of the surfaces were observed using a scanning electron microscope (SEM) (JSM 7001F; JEOL, Ltd., Japan) with energy dispersive spectroscopy (EDS) (Oxford Inca Energy-350; Oxford Instruments plc). Different phases formed in the alloyed layers and FWHM of presented peaks were identified by X-ray diffractometer (D8 ADVANCE; Bruker Corporation, Germany) with Cu Kα radiation. The 2θ was in the range of 30° to 120° with a scanning speed of 1°/min.

2.3 Microhardness analysis

The microhardness distribution in the cross section of the alloyed layer was measured using Vickers indenter (HXD-1000TMSC/LCD, Shanghai Taiming Optical Instrument Co. Ltd., China) at a load of 50g and a dwell time of 10s. The distance between indentations was in the range of 30-50 μm.

2.4 Wear experiments

Dry sliding wear test of the tempered layer and substrate was performed with a ball-on-disk apparatus (CETR UMT-2 tribometer) in ambient air using the straight line reciprocating wear form. Their surfaces were grounded and polished with 1200 grit paper and rinsed with alcohol before wear testing. The counter-body was a Co-WC hard alloy ball with a diameter of 9.5 mm. The applied load was 20 N. The workbench was set to rotate at a fixed speed of 300 rpm and the test time was 60 minutes. The reciprocating length of the wear track was 5 mm. Friction coefficient was recorded. The wear ball was changed prior to each test to ensure reproducibility in the wear conditions. The contour curves of the cross-section of the wear tracks were measured on 5 different positions for each specimen with a Surface Texture Measuring Instrument (SURFCOM 130A, ACCRETECH, Tokyo, Japan). The data measured was processed to get the area value through integrating with software Origin 8.0. The average of the five groups of area data was taken as the final value for each specimen. The wear volume loss is the product of the cross-section area and length of the wear track, which was used to evaluate the wear resistance. The microstructure of top surface of the wear track was observed with SEM.

3. Results and discussion

3.1 Phase and FWHM analysis

X-ray diffraction of the alloyed and tempered layer is shown in Fig. 1. Both layers show the presence of M23C6 and iron carbides (Fe3(C, B), Fe3C). Differences are that Fe3(C, B) and residual austenite changed to Fe3C and ferrite in the tempered layer. A point worth mentioning is that martensite is not
observed in the alloyed layer by XRD, due to the small volume fraction of it. A lot of hypereutectic carbides form initially from the liquid, followed by the eutectic reaction with the formation of ledeburite. Small proportion of austenite attributes to the small amount of transformed martensite. Residual austenite was always observed in laser treated steel layers or coatings due to the fast cooling rate [1, 11, 12]. Laser alloying is a fast heating and cooling process. Under non-equilibrium solidification, metastable phases are easily formed, such as the formation of M23C6 and Fe3(C, B), as the composition and thermal condition (gradient, cooling rate) of different areas varied enormously. Atoms (C, B) absorbing the energy offered by tempering started to diffuse, and stoichiometric carbide was formed in the tempered layer. Furthermore, residual austenite transformed into ferrite after a series of transformation. First, carbon in the martensite segregated. Second, martensite transformed into tempered martensite, with the precipitated tiny flaky carbides distributing in the martensite matrix. Third, residual austenite decomposed. Fourth, epsilon carbide dissolved into $\alpha$ phase and at the same time, cementite precipitated from $\alpha$ phase. Solubility of carbon in martensite decreased to the equilibrium composition of ferrite. Tempered martensite transformed into tempered troostite, with fine granular cementite distributing in ferrite. Fifth, cementites aggregated and grew up and ferrite recrystallized. These elaborations explained the existence of ferrite in the tempered layer.

The X-ray measurements allowed researchers to obtain important information about the surface state of material: the width of the diffraction peak at half the maximum value of diffraction (FWHM). This quantity is related to the grain distortion, to the dislocation density and to the so called type II micro residual stresses [13]. It is assumed as an index of the hardening of the material, which indicates that, the larger the FWHM is, the harder the measured surface. FWHM values of the first primary peak in the alloyed and tempered layer were 0.5196 and 0.2558 with the 2 theta position at 43.4832 and 44.8195 deg. Peak positions shifted to a relatively large Bragg’s angle after tempering, indicating a decrease in lattice parameters. Narrowing of diffraction peaks testified a decrease of defects in structure and high strain values after laser treatment [14]. Furthermore, dense dislocations were reported in broadened austenite in laser melted layer. The narrowing of diffraction peaks testified a decrease of defects in structure and high strain values after tempering. This confirmed that stress release reduced the solid solubility of dissolved alloying elements kept in the solid solution after laser alloying due to the precipitation of carbides. Colaco et al reported a reduction of the super-saturation of martensite induced by post heat treatment followed by laser surface melting [15], which was similar to our results.

### 3.2 Microstructure analysis

Microstructure of the tempered layers is shown in Fig. 2. It is composed of hypereutectic carbides and eutectic ledeburite. However, compared with those in Fig. 2 [10], the heat and oxidation effect are observable.

![Figure 1. XRD pattern of (a) alloyed layer, (b) tempered layer.](image_url)
3.3 Microhardness analysis

Microhardness distribution in the tempered layer is shown in Fig. 3. The average value decreased from 1001 HV0.05 of the alloyed layer to 558 HV0.05 of the tempered layer, decreasing by 44%. The average value decreases from 656 HV0.05 of the roll substrate to 410 HV0.05 of the tempered substrate, decreasing by 37.5%. Heat treatment decreased the surface hardness of the shot-peened nitride steel due to the residual stress release [16]. Tempering decreased residual stress and hence the microhardness in this investigation. In addition, diffusion of carbon and precipitation of carbides decreases the degree of the super-saturation of solid solution, resulting in the decrease of microhardness.

3.4 Friction and wear behavior

Friction coefficients of the tempered layer and substrate were close after sliding for 3600s as shown in Fig. 4. The running-in time for the tempered layer and substrate were 80s and 300s, respectively. The running-in wear mechanisms were very complicated, simultaneously accompanied by plough, adhesive, fatigue and oxidative wear, etc. It was fast for the tempered layer to run into steady wear process.
Contour curves of the cross-section of the wear tracks of the tempered layer and substrate are shown in Fig. 5. Wear volume losses were 1.735 ×10-3 and 1.92×10⁻³ mm³, respectively. Relative wear resistance of the tempered layer was 1.11 times that of the substrate.

Worn surface morphology of the tempered layer is shown in Fig. 6(a) and (b). Thick oxide films covered the tempered layer. No ploughing grooves can be seen on it. However, spalling of oxide films were observed due to the shear force during wear process. Shallow ploughing grooves, detached wear debris, and shrouded oxide films can be seen on the worn surface of tempered substrate shown in Fig. 6(c) and (d), indicating abrasive and adhesive wear. The surface films were the results of complex physical and chemical processes, including material transfer, debris generation and oxidation. These films have unique lubrication and electrical properties [17, 18]. The temperature rise caused by heating increased the wear of the material and favors oxidation [19, 20]. These oxide films served as the interface between the friction pair, avoiding the formation of large friction heat and energy between two sliding metallic components. However, the integrity and the bonding strength with the matrix of oxide films was one important factor to determine the wear resistance of matrix [21].

4. Conclusions

Effect of tempering at 500°C on the microstructure, phases, FWHM of peaks, microhardness and wear
behavior of laser alloyed layer on high-Ni-Cr infinite chilled cast iron roll was investigated. The conclusions are as follows:

1. Ferrite was formed in the tempered layer.
2. Metastable carbides changed to stoichiometric carbides after tempering.
3. FWHM (full width at half maximum) of the first primary peak was decreased by tempering.
4. Microhardness decreased by 44%.
5. Friction coefficient of the tempered layer exhibited gentle increase and a stable value of 0.6 was achieved for both the tempered layer and substrate.
6. Relative wear resistance of the tempered layer was 1.11 times that of the tempered roll substrate.
7. Stress release, decreased solid solubility, improved toughness and easy formation of oxide films were positive to the wear resistance of the tempered layer.

Acknowledgments

Financial supports from the National Science Foundation of China (No. 51201070), Doctoral Fund of Ministry of Education of China (No. 20113227120006), Natural Science Foundation of Jiangsu Province of China (No. BK2012713), Special Research Foundation of Young teachers of Jiangsu University of Science and Technology (112110122) and the University Science Research Project of Jiangsu Province (11KJB460001) are acknowledged.

References

[1] Sun G F, Zhang Y K, Liu C S, Luo K Y, Tao X Q and Li P 2010 Mater. Des. 31 2737-44
[2] Sun G F, Zhou R, Li P, Feng A X and Zhang Y K 2011 Surf. Coat. Technol. 205 2747-54
[3] Zeinberger K H and Windhager M 2003 45th MWSP Conf. Proc. (Warrendale: Iron & Steel Society) P133-42
[4] Masanta M, Shariff S M and Roy Choudhury A 2011 Wear 271 1124
[5] Jagdheesh R, Murali U K and Nath A K 2007 Surf. Eng. 23 93-8
[6] Sharma G, Awasthi R and Chandra K 2010 Intermetallics 18 2124-27
[7] Klachuk S and Bamberger M 2010 Mater. Sci. Tech. 26 1059-67
[8] Xin X Z, Chen J, Xiang N, Gong Y and Wei B 2014 Dent. Mater. 30 263-70
[9] Takaichi A, Suyalatu, Nakamoto T, Joko N, Nomura N, Tsutsumi Y, Migita S, Doi H, Kurosu S, Chiba A, Wakabayashi N, Igarashi Y, Hanawa T 2013 J. Mech. Behav. Biomed. 21 67-76
[10] Sun G F, Liu C S, Tao X Q and Chen S Y 2008 J. Northeast. Univ. (Nat. Sci.) 29 845-8
[11] Colaco R and Vilar R 1998 J. Mater. Sci. Lett. 17 563-7
[12] Colaco R and Vilar R 2005 Wear 258 225-31
[13] Noyan I C and Chen J B. 1987 Residual Stress Measurement by Diffraction and Interpretation (New York: Springer-Verlag)
[14] Burg M Van Den and Hosson J Th M De 1993 Acta Metall. Mater. 41 2557-64
[15] Colaco R, Gordo E, Ruiz-Navas EM, Otasevic M and Vilar R 2006 Wear 260 949-56
[16] Pariente I F and Guagliano M 2008 Surf. Coat. Technol. 202 3072-80
[17] Zaidi H, Chin K J and Frene J 2001 Surf. Coat. Technol. 148 241-50
[18] Hu Z L, Chen Z H and Xia J T 2008 Wear 264:11-7
[19] Ding T, Chen G X, Zhu M H, Zhang W H and Zhou Z R 2009 Wear 267 1080-6
[20] Yi F, Zhang M and Xu Y 2005 Carbon 43 2685-92
[21] Zhang Q, Jiang Z Y, Wei D B, Zhu H T, Chen Z X, Han J T and Xie G L 2013 Wear 301 598-607