THERMAL STABILITY OF LASER TREATED DIE MATERIAL FOR SEMI-SOLID METAL FORMING

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ABSTRACT: This paper presents laser surface modification work performed to improve the lifetime of die materials. Die material AISI H13, with typical hardness in the range of 42 to 48 HRC, offers high wear and corrosion resistance. However the cyclic high temperature conditions along with exposure to high viscosity molten metal in semi-solid forming cause the die to wear and crack with resultant shortened die lifetime. In this study, the thermal stability of die material at elevated temperature was investigated through micro-hardness testing and a metallographic study. AISI H13 samples were laser glazed using CO₂ continuous wave mode laser with 10.6 µm wavelength. Samples were attached to a specially designed rotating chuck to enable it to be rotated at speeds up to 1500 rpm and allow flat surface glazing to take place. The microhardness was measured for as-glazed samples and annealed samples which were held at temperatures ranging from 550°C to 800°C with 50°C intervals. The metallographic study conducted examined the formation of three zones at different depths which were the glazed zone, the heat affected zone and the substrate. As a result of rapid heating and cooling from the laser glazing process, a metallic glass layer was developed which exhibited an average micro-hardness of 900 HV when exposed to 3.34E+10 W/m² laser irradiance within a range of 0.0011 to 0.0018 s exposure time. Crystallization in glazed zone increased as the annealing temperature increased. As the annealing temperature reached above approximately 600°C, the micro-hardness decreased to approximately 600 HV (equivalent to approx. 54 HRC) due to local crystallization. These findings show potential direct application of glazed dies for non-ferrous semi-solid forming and the requirement for thermal barrier protection for application at higher temperatures.

KEYWORDS: Laser glazing, thermal stability, micro-hardness, annealing and die material.

1 INTRODUCTION

Laser glazing has been studied as a way of surface hardening on a die, in order to overcome the premature failure of dies in semi solid casting, by developing an amorphous layer on the surface. Coating technologies are currently used to protect tooling surfaces. With these it is difficult to meet the six requirements of effective coating; excellent bonding, adequate thickness, absence of flaws, suitable mechanical properties, thermal shock resistance and high temperature stability [1].

In semi-solid processing, the forming temperatures are considerably lower than in liquid metal die-casting, which can extend die life. Die material AISI H13, with typical hardness in the range of 40 to 50 HRC, offers high wear and corrosion resistance [2]. The cyclic high temperature conditions along with exposure to high viscosity molten metal in semi-solid forming would cause the die to wear and crack with resultant shortened die lifetime. Though H13 steel is able to withstand relatively high working temperatures above 600°C the die easily wears such that die life at high temperature is not sufficiently long [3].

In previous work, a laser glazed steel surface exhibited hardness enhanced by as much as 30% compared to the substrate. This was as a result of fine grains and secondary carbide formation [1]. Other studies on laser glazing indicate that a thin layer of laser glazed surface can possess hard non-equilibrium microstructures that are intimately bonded to the substrate and the modified region itself [4]. A number of phases were present after the process due to rapid solidification and rapid cooling. However, referring to Yang et al., an amorphous layer was developed from the laser glazing process [5]. The layer occurred even in multi pass regions where overlapped zones did not recrystallize during the subsequent laser pass. To improve the die lifetime, surface modification using laser glazing was investigated in this work. The thermal stability of die material at elevated temperature was investigated through micro-hardness testing and metallographic study. The combination of wear resistance, high strength and thermal stability can increase significantly the lifetime of the die and therefore decrease unit production cost.

2 EXPERIMENTAL

The AISI H13 samples processed and examined in this study were 40 mm diameter and 50 mm long cylindrical specimens. The flat 40 mm diameter surface was processed by rotating it perpendicular to the laser beam firing direction as shown in Figure 1. The sample was also linearly translated in order to glaze a pre-defined surface area.

The laser was started at the edge of the sample, at a radius of 20 mm, and stopped prior to the centre point to avoid sample vaporisation due to excessive energy density.

This laser surface glazing was performed on the as received material. Chemical composition of AISI H13 measured with an energy-dispersive X-ray spectrometer is shown in Table 1.

Samples were glazed by continuous operating mode of the CO_2 laser. The laser system specifications are detailed in Table 2. To examine the range of exposure times and

Table 1: Chemical composition of AISI H13

power densities that are referred to in previous works [4-8], the rotation speed was kept constant at 1500 rpm. This resulted in increased exposure time as the laser spot radius decreased as the linear translation speed was kept constant. After laser glazing the samples were also annealed at temperatures of 550°C, 600°C, 650°C, 700°C, 750°C and 800°C. Samples were annealed for 15 minutes and cooled in ambient air to observe the effects of elevated temperatures on the laser glazed surface properties including the structure and hardness. Samples were sectioned and metallographic preparation was done through grinding, polishing and nital (2-10%) etching. Formation of a glazed layer, including grain size measurement, was examined with an EVO-LS15 Scanning Electron Microscope (SEM). The micro-hardness properties on the laser glazed surface and annealed samples was measured using Vickers test at a set load of 981 mN.

Element	С	Mn	Si	Cr	Ni	Мо	V	Cu	Р	S	Fe
wt %	0.32-0.45	0.20-0.50	0.80-1.20	4.75-5.50	0.30	1.10-1.75	0.80-1.20	0.25	0.03	0.03	balance



Figure 1: Laser glazing apparatus

Table 2: Laser system specifications

Power	1.2 kW
Beam geometry	Circle
Focal position	Surface
Spot size	0.2 mm
Assist gas	Argon
Laser beam mode	TEM_{00}
Laser wavelength	10.6 µm

3 RESULTS AND DISCUSSION

3.1 EXPOSURE TIME – STRUCTURE RELATION

Grain size development in the laser glazing process depends on the local cooling rate. In the absence of highly sophisticated thermal model or thermal image camera to determine the temperature gradient, the cooling rate is most often estimated by calculating the exposure time. Due to change in laser spot surface traverse speed through the process, a range of exposure times resulted in variations in microstructure formation.

Figure 2 shows an SEM back scattered detector micrograph of as received H13 tool steel. From the micrograph, it can be seen that the as received H13 tool steel grain size was between approximately 3 and 9 μ m.



Figure 2: SEM back-scattered detector micrographs of asreceived H13



Figure 3: SEM back-scattered detector micrographs of grain size in laser glazed tool steel taken parallel to the surface with an exposure time of (a)0.0018 s, (b) 0.0011 s, (c)0.00059, and (d)0.00029 s.

The micrographs in Figure 3 (a) and (b) show amorphous material which was found at radial distances of 3.5mm and

4.7 mm respectively from the centre of the flat circular surface (see Figure 1). Figure 3 (c) shows the largest grain size recorded at a radial distance of 5.2 mm. Figure 3 (d) shows the microstructure with nano-grains at a radial distance of 11 mm. These macrographs were taken in the region of the flat surface which had locally melted. The amorphous structure in Figure 3(a) and (b) resulted from exposure times of 0.0018 s and 0.0011 s respectively. Micro-sized grains were observed in Figure 3(c) as a result of a 0.00059 s exposure time and nano-sized grains were observed in Figure 3(d) which corresponded to an exposure time of 0.00029 s. A longer exposure time developed large porosity defects as pointed to in Figure 3(a). Micrographs in Figures (a) through (c) were taken from a relatively rough region of the processed surface. The high energy densities with the relatively low exposure times resulted in material ejection from the surface leaving small scribed circular markings. A larger amount of material removal was seen in the regions of Figure 3 (a) and (b) compared to Figure 3 (c). The larger grains in figure 3 (c) were potentially due to this lower amount of material removal where local heat buildup caused a slower local cooling rate. Interestingly, the local cooling rate in the region of Figure 3 (d) produced a smaller grain size. An optimum set of processing conditions can be sought, close to those produced at this position, which does not involve material removal but results in sufficiently high cooling rates.

3.2 MICROHARDNESS OF LASER GLAZED H13

As a result of rapid heating and cooling, a treated layer was developed on the tool steel surface which exhibited an average micro-hardness of 900 HV (equivalent to approximately 67 HRC). It can be seen in Figure 4, an increase in hardness from 800 HV to 1200 HV were achieved in the treated surface. At room temperature, 1200 HV is the maximum hardness that was achieved within the treated region. Then the hardness value start decreasing from 1200 HV to 300 HV when reaching deeper into the substrate. 300 HV is the hardness for the as received H13 tool steel. Understanding the micro-mechanisms of the crystallization to impede or control crystallization is therefore prerequisite for most applications, as the stability against crystallization determines the effective working limits [8].

3.3 THERMAL INSTABILITY OF LASER GLAZED SURFACE

A striking increase of the surface hardness is significant for wear and elevated temperature applications. The amorphous material is however instable when sufficient temperature is applied. This is due to crystallization of the amorphous layer at various annealing temperatures which causes changes in micro-hardness.



Figure 4: Microhardness of glazed layer at room temperature

Figure 5 indicates that as the annealing temperature reached above approximately 600°C, the micro-hardness decreased to approximately 600 HV (equivalent to approx. 54 HRC) due to local crystallization. Basically, the crystallization in the glazed zone increased as the annealing temperature increased.



Figure 5: Microhardness as a function of the distance from surface at different annealing temperature

Parallel with previous works of Nieh and Wadsworth, the changes of mechanical properties in amorphous metal are characterized by inhomogeneous and homogenous deformation at low and high temperature respectively [10]. Due to consistency of the crystal structure produced at annealing temperatures of 750°C and 800°C, there was little variation in micro-hardness with depth as shown in Figure 5 The micro-hardness of the laser glazed layer was approximately 250HV along the depth. These results indicate that a gradual transformation from an amorphous structure to a crystalline structure occurred between 600°C and 750°C. A corresponding increase in grain size was noted with this transformation. At holding temperatures below 800°C and above 600°C, some degree of retention of the original higher micro-hardness values was noted. This

was most likely due to the metastable structure of the laser glazed surface.

4 CONCLUSION

This study is beneficial in pointing out the effective working limit (approx. 600°C) for laser glazed die surface through observation and measurement of surface structure and hardness. These findings also show potential direct application of glazed dies for non-ferrous semi-solid forming and the requirement for thermal barrier protection for application at higher temperatures.

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