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Citation: Aqida, S.N., Brabazon, D. & Naher, S. (2012). Atomic diffusion in laser surface modified AISI H13 steel. Applied Physics A: Materials Science & Processing, 112(1), pp. 139-142. doi: 10.1007/s00339-012-7214-x

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Applied Physics A: Materials Science and Engineering, 2012

Appl Phys A
DOI 10.1007/s00339-012-7214-x

Atomic diffusion in laser surface modified AISI H13 steel

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Abstract This paper presents a laser surface modification process of AISI H13 steel using 0.09 and 0.4 mm of laser spot sizes with an aim to increase surface hardness and investigate elements diffusion in laser modified surface. A Rofin DC-015 diffusion-cooled CO2 slab laser was used to process AISI H13 steel samples. Samples of 10 mm diameter were sectioned to 100 mm length in order to process a predefined circumferential area. The parameters selected for examination were laser peak power, pulse repetition frequency (PRF), and overlap percentage. The hardness properties were tested at 981 mN force. Metallographic study and energy dispersive X-ray spectroscopy (EDXS) were performed to observe presence of elements and their distribution in the sample surface. Maximum hardness achieved in the modified surface was 1017 HV0.1. Change of elements composition in the modified layer region was detected in the laser modified samples. Diffusion possibly occurred for C, Cr, Cu, Ni, and S elements. The potential found for increase in surface hardness represents an important method to sustain tooling life. The EDXS findings signify understanding of processing parameters effect on the modified surface composition.

1 Introduction

Laser surface modification can be used to enhance tribological and mechanical properties, including hardness, strength, toughness, fatigue strength, and corrosion resistance [1]. In laser processing, control of parameter settings yields different effects on the modified surface properties. The laser power and scan speed or material-laser interaction time were observed to have a strong influence on the resulted temperature profile and modified layer depth [2, 3]. Increasing the laser power increases the surface temperature, which influences the molten surface solidification rate. While a high cooling rate is necessary to refine the grain structures, a high power density and short interaction time combination is generally preferred to allow for a faster cooling rate to be achieved.

Heating and cooling rates in laser surface melting are both significant to determine the mechanical properties of the modified surface. Each laser processing applies different rates. Despite the fact that laser processing offers rapid heating and cooling, a few milliseconds faster or slower can result in very different grain sizes, microstructures, and phases. Molten surfaces experience undercooling where the cooling occurs below the transformation temperature and without phase transformation. Pulse energy affect surface temperature where the surface experiences a higher temperature when processed with a short pulse in contrast to longer pulses due to high power intensity of the short pulse [4]. Short pulses result in high fluence which affects the heating and cooling rate. Results from solidification theory indicate that cooling rate is reduced as the fluence was increased. At lower fluence, the cooling rate was higher and caused

more undercooling [5]. In thermomechanical processing, undercooled austenite transforms to ultrafine ferrite ranging from 1 to 3 μm or less with enhanced strength and ductility [6]. Undercooled metallic melt experienced non-equilibrium solidification, formation of metastable phases as well as atomic diffusion [7–9]. Variation of heating and cooling rate influenced atomic diffusion rate. Though much works have been conducted on the atomic diffusion of laser processed surface, but little information found on the effect of laser spot size and processing parameters variation. In this paper, the effect of various laser processing parameters on atomic diffusion of laser modified H13 surface was investigated. Enhancement of average hardness measured in laser modified surface was highlighted.

Table 1 Laser processing parameters

Set parameters	Sample						
	F1	F2	F3	F4	E1	E2	E3
P_p (W)	760	1515	760	1138	760	760	760
DC (%)	36	18	36	24	85	100	100
η (%)	0	10	0	10	10	30	50
PRF (Hz)	3500	2900	2300	2300	2857	3810	4000
Spot size (mm)	0.09					0.4	
Outcome parameters							
I (W/mm^2)	119,459	216,449	119,297	162,398	5,548	4,631	3,971
T_R (ms)	0.10	0.07	0.16	0.12	0.32	0.34	0.38
E_p (J)	0.08	0.10	0.12	0.12	0.23	0.20	0.19

2 Experimental

The material investigated in this study was AISI H13 die steel with chemical composition of 0.32–0.45 wt% C, 0.20–0.50 wt% Mn, 0.80–1.20 wt% Si, 4.75–5.50 wt% Cr, 0.3 wt% Ni, 1.10–1.75 wt%Mo, 0.80–1.20 wt% V, 0.25 wt% Cu, 0.03 wt% P and S, and the balance content for Fe. These compositions were measured using spark light emission spectroscopy. As-received 10 mm diameter H13 die steel cylindrical rods were sectioned into 100 mm length samples. The samples were cleaned with ethanol prior to processing. Three samples of E1, E2, and E3 were chemically etched prior to processing. For sample F1, F2, F3, and F4 sample surface was laser processed only after surface roughening to produce 3.0±0.2 μm average roughness and then chemically etched using nital solution (8–10 %). A Rofin DC-015 diffusion-cooled CO2 slab laser system with 10.6 μm wavelength was used to modify the sample surface. The laser system was focused to a minimum laser spot diameter of 90 μm onto the sample surface. A larger spot size diameter of 0.4 mm was obtained by defocusing the laser beam below the sample surface. Table 1 shows the parameter settings used to process the samples. The outcome parameters from the settings

were residence time, *TR*, irradiance, *I* , and pulse energy, *Ep*. The modified layer and as-received substrate hardness properties were measured using Leitz miniload tester with 981 mN force. The hardness property was determined by Vickers hardness scale (HV0.1). The Cross section of the sample with indentation marks was captured using Reichart ME F2 microscope. The chemical composition of the samples was measured quantitatively using Oxford Instruments-Inca *x*-act and microanalysis suit EDXS. The samples were observed using an EVO-LS15, Carl Zeiss, scanning electron microscope (SEM) while the EDXS detector was used to collect composition data from the sample. Identification of elements on the sample cross-sectional surfaces was carried out using the integrated software.

3 Results and discussion

The as-received H13 steel substrate hardness was 280 HV0.1 while the modified surface average hardness measured from samples processed using 0.09 mm spot size F1, F2, F3, and F4 was 821, 816, 868, and 871 HV0.1, respectively. The indentation marks in Fig. 1 were observed on the sample F4 cross section where a smaller mark was obtained in the modified surface region compared to the marks measured in the substrate. For all samples processed at 0.4 mm spot size, the average hardness measured was 1017 HV0.1. The hardness enhancement in the modified layer of more than three times the substrate was due to rapidly melted and solidified H13 surface. Rapid solidification yields smaller grain size that exhibit high hardness. From Fig. 1, smaller grain size in modified layer resulted in small indentation mark which exhibit higher hardness. The resulting chemical composition of the H13 steel substrate and laser modified samples analyzed using EDXS is given in Table 2. In Table 2, ten elements were detected from the H13 steel substrate; carbon (C), silicon (Si), sulphur (S), vanadium (V), chromium (Cr), manganese (Mn), iron (Fe), nickel (Ni), copper (Cu), and molybdenum (Mo). The chemical composition of laser processed H13 tool steel samples was compared with the H13 tool steel substrate. The analysis for E1 sample was conducted using a single spectrum where the chemical composition was measured from the area selected. Samples E2 and E3 were analyzed using line spectra. The resulting chemical composition of the modified layers is given in Table 2. The presence of eight elements in sample E3 was detected while in sample E2, nine elements were identified. In sample E2, the content of C was higher; while Cr content was lower when compared with sample E3. Cu was detected in sample E2 at 0.24 wt% which was absent in sample E1 and E3. In sample E1, Mo was detected at 1.46 wt%. Cr was recorded at is the highest level in sample E3 among the three samples. Variations of elements composition were observed where the samples were processed at different overlap, PRF and pulse energy. The maximum range of C in sample E2 was higher than the maximum level detected in sample E3. In Table 2, sample F1, F2, F3, and F4 were analyzed using line spectra. In F2 and F3 samples, seven elements were detected, while eight elements were analyzed in F1 and F4 samples. In sample F4, C, V, Cr, Mn, Fe, Si, S, and Mo were detected. Two elements, Cu and Ni, were absent in all four samples processed at 0.09 mm spot size. The processing parameter seemed to vary the elements composition where S was detected in sample F4 and F1 but was none in sample F3 and F2. Cr and Mo composition was the highest in sample F2 compared to other samples observed. Figure 2(a) shows a back-scatter detector SEM micrograph with area selection in the H13 steel substrate used for compositional area analysis. In the micrograph of sample E1, distribution of elements like Ni, Mo, and V resembled the grain boundaries geometries. An image of Ni distribution that shows its presence in grain boundaries is given in Fig. 2(b).

The EDXS results showed that higher carbon percentage was detected in the as-received H13 tool steel when compared to the composition measured by spark light emission spectroscopy. EDXS is commonly known however to pro- duce C concentration results than are actually present due

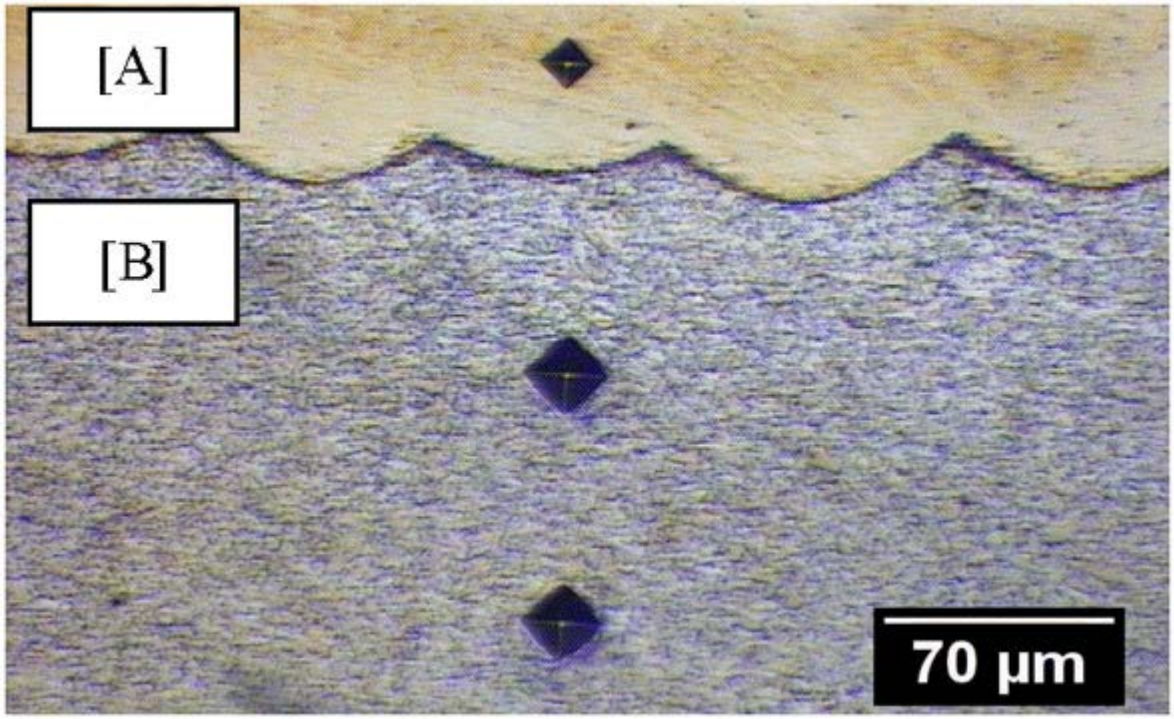

Fig. 1 Increase of indentation diameter from modified surface [**A**] to substrate material [**B**] in sample F4

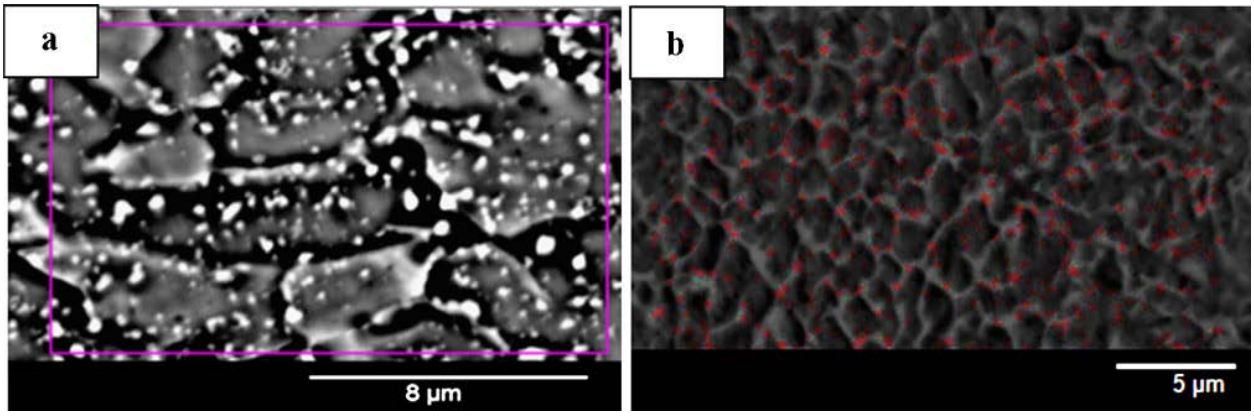

Fig. 2 Back scatter detector SEM micrograph of (a) selected area in AISI H13 steel substrate, and (b) nickel elements distribution in sample E1

Table 2 Chemical composition of the H13 tool steel substrate and laser modified samples

Elements (wt%)	C	Si	S	V	Cr	Mn	Fe	Ni	Cu	Mo
H13 substrate	1.56	1.13	0.47	1.10	5.01	0.42	88.41	0.18	0.21	1.51
Sample E1	5.47	1.12	–	0.94	4.98	0.46	85.35	0.22	–	1.46
Sample E2	7.05–12.11	0.87–1.21	0.35–0.44	0.80–0.95	4.59–4.93	0.42–0.49	81.19–84.99	0.20–0.22	–	–
Sample E3	5.31–9.05	0.94–1.33	0.35–0.89	0.77–1.33	4.44–6.23	0.44–0.62	82.85–86.77	0.26–0.31	–	–
Sample F1	0.30–6.76	1.13–1.22	0.49–0.50	0.89–0.99	4.69–5.26	0.42–0.48	85.80–90.90	–	–	1.46
Sample F2	1.25	1.24–1.27	–	0.97–1.27	5.27–6.23	0.46–0.48	88.58–92.02	–	–	1.50–2.18
Sample F3	0.01–4.67	1.24–1.41	–	0.95–1.45	4.98–5.53	0.46–0.48	87.35–89.72	–	–	1.73–1.79
Sample F4	0.33–4.97	1.11–1.29	0.48–0.52	0.82–1.04	4.99–5.44	0.44–0.46	87.15–91.03	–	–	1.52

to the lack sensitivity of the lower mass elements. In the EDXS analysis, the untreated H13 tool steel cross section was measured from substrate region of laser modified sample. The high rate of heating and cooling during processing had possibly increased the percentage of C through diffusion. However, as the measurements were carried out deeper into the substrate, the cooling rate was much slower than the processed surface which explains the uniform distribution of elements in the selected area.

The parameters used in modifying the H13 tool steel samples caused variation of elements composition. By heating the sample surface at its liquidus temperature of 1454 °C, the elements were redistributed. From the analysis conducted for samples E3, E2, and E1 which were processed at pulse energies of 0.19, 0.20, and 0.23 J, the existence of the elements like Mo, Cu, and Ni, and the range of the elements composition in the modified layer were determined. Diffusion of elements occurred during laser heating as the modified layer composition changed from that of the H13 tool steel substrate. Diffusion possibly occurred for C, Cr, Cu, and S elements. The C diffusion was identified in the three samples (E1, E2, and E3) as the C content was higher when compared to the EDXS measurements of the H13 tool steel substrate. Increased pulse energy increased the sample temperature which increased the atomic diffusion rate. For sample E1, the high pulse energy of 0.23 J had resulted in a high diffusion rate in the molten surface. However under a high cooling rate, the rapid decrease of melt temperature trapped the atoms in their location. It seemed that the presence of elements in the modified layer was dependant on the energy, residence time, and speed of undercooling during processing. The effect of higher laser scan speed on lowering the elements diffusion in molten region was also reported in previous works [10].

In samples processed at 0.09 mm spot size, the higher irradiance settings with 0.08, 0.10, and 0.12 J pulse energy had caused more diffused elements in the modified layer. In samples F3 and F2, with the range of cooling rates 5.06 × 106 to 5.09 × 106 K/s, sulphur diffusion occurred; see Table 2. Sulphur diffuses in steels at high temperatures (more than 1000 °C). A similar finding was reported previously [11]. For all four samples processed at 0.09 mm spot size, Ni diffusion possibly occurred in the modified layer due to the higher irradiance used during processing compared to the three samples processed at 0.4 mm spot size. In general, Ni diffusion in steel occurs at high temperature (over 1100 °C) [12]. Ni diffusion was observed in all four samples (F1, F2, F3, and F4) where the irradiances were between 119,297 and 216,449 W/mm2. These irradiances increased the sample surface temperature rapidly at shorter residence times compared to the lower irradiances and longer residence times in samples E1, E2, and E3. The

shorter residence time in sample F8 produced a higher composition of Cr and Mo elements in the modified layer when compared to other three samples. Shorter residence time increased the undercooling speed which possibly stopped the diffusion earlier than in other samples.

4 Conclusion

Hardness enhancement in modified samples measured was more than three times the untreated H13 steel substrate. Change of elements composition in the modified layer region was detected in laser modified samples. The surface composition variation was due to the processing parameters: irradiance and residence time. Heating the samples at high temperature allowed atomic diffusion to occur. The rapid drop of temperature provided the stopped further atomic diffusion and then froze the present structure in place. High irradiance and short residence time in samples processed using 0.09 mm spot size caused a great degree of element diffusion compared to samples processed by 0.4 mm spot size.

Acknowledgements The authors would like to acknowledge the support from the Ministry of Higher Education Malaysia, Universiti Malaysia Pahang, and Dublin City University for funding this research.

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