Fracture Microindentation on boride layers on AISI 1020 steel

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Abstract. In this paper, an attempt has been made to enhance the fracture toughness (Kc) of boride layer using multi-component (Ni, Cr and B) laser boriding. The fracture toughness of continuously pack borided, interrupted pack borided and multi-component (Ni, Cr and B) laser borided steel specimens was measured using Vickers microindentation fracture toughness test as per ASTM E384 standard. The fracture toughness of continuously pack borided layer was 2.5 – 3.3 MPa.m¹/². The fracture toughness of interrupted boride layer was in the range of 3.5 – 4.9 MPa.m¹/². The fracture toughness of multi-component (Ni, Cr and B) laser borided layer was in the range of 13.8 – 18.3 MPa.m¹/². A significant improvement in fracture toughness of laser treated specimens was observed from the experimental results. This may be due to better distribution of boron, nickel, chromium and other alloying elements due to laser treatment and relatively more uniform boride layer as compared with continuously pack borided layer and interrupted pack borided layer.

1. Introduction

High hardness, high wear resistance and good chemical stability of the boride layers resulting in the increased service life of steel components, have long attracted the attention of research metallurgists and manufacturers [1]. During thermo-chemical boriding of plain carbon steels, very high brittleness is generated in the boride layer as well as in the core [2]. High brittleness is reported due to the presence of two phase (Fe₂B-FeB) microstructure especially due to hard and brittle FeB phase (formed due to high boron potential), lateral cracks at FeB/Fe₂B interface, non-uniform distribution of alloying elements in the boride layer and long time exposure (1 to 12 h) of the steel at elevated temperature (above 950 °C) resulting in grain coarsening at the core [3]. Because of this brittleness problem, widespread industrial acceptance of borided steels has been limited even though it has a very high surface hardness in the range of 1400-2100 HV [4]. The task of minimising the brittleness of the boride layers without much compromising the surface hardness is a challenging problem. Various methods have been proposed by the researchers to improve the properties of boride layer. Diffusion annealing of an as-borided steels, by heating the as-borided steels at 950 °C for 1 to several hours, to obtain single phase Fe₂B [5]. Multi-component diffusion of steel with alloying elements such as chromium, nickel, aluminium along with boron with an aim to reduce the boron concentration in the boride layer and thereby the mechanical properties of the boride layer may be improved, [6]. Interrupted boriding, which produces single phase (Fe₂B), more rounded, uniform, thicker and shorter boride needles resulting in better toughness of the boride layer [7]. Laser surface modification of borided steels
results in breaking down of the acicular microstructure at the surface to achieve globular microstructure, can substantially improve the boride layer toughness [8]. Superplastic boronising, which produces a non-acicular boride structure with equiaxed grains of the borides thereby reducing the brittleness [9]. Boriding by surface melting methods such as ion beam [10], electron beam [11], plasma transferred arc boriding [12] and laser beam [13] which produces hard and thick boride layers on steels without much disturbance to the bulk properties of the steels. The focus of the present research is to develop a boride layer on AISI 1020 steel to get adequate hardness (1300 – 1500 HV) and improved fracture toughness of the boride layer without much disturbing the bulk properties of the steel. Hence, multi-component (Ni, Cr and B) laser boriding has been attempted. First a layer of nickel (Ni) and then another layer of chromium (Cr) were electroplated on the surface of steel. Then a paste of boron carbide (B₄C) was applied and subsequently laser treated at different energy densities.

2. Experimental procedure

2.1. Material
A low carbon low alloy steel (0.23% C, 0.33% Mn, balance% Fe) was used as a base material (AISI 1020) for boriding. The steel specimens were normalized before boriding. The steel had a hardness of 119±5 HV (at 50 g load). The specimen dimensions for optical, microhardness and fracture toughness studies were 20 mm (length) x 20 mm (breadth) x 5 mm (thickness).

2.2. Continuous pack boriding
Continuous pack boriding was carried out on AISI 1020 steel using boron carbide based paste process in a muffle furnace at 950 °C for 3 h. More details of the experimental procedure are described elsewhere [8].

2.3. Interrupted pack boriding
Interrupted pack boriding is a modified pack boriding process [8]. The packing procedure is similar to continuous pack boriding, but the difference is in the heat treatment cycle of the process. The specimens were subjected to heating at 950 °C for 1 h. After 1 h of boriding, the stainless steel box containing the boriding mixture and specimens were removed from the furnace and allowed to cool in still air for 45 min and then it was loaded in the muffle furnace. This procedure was repeated three times.

2.4. Multi-component (Ni, Cr and B) boriding
This is a three stage process. First Ni and then Cr were electroplated on the surface of the AISI 1020 steel as two different layers one over the other. Then a paste of B₄C was applied on the as-plated surface and subsequently laser boriding was done at two different energy densities i.e., 47 x 10⁶ J/m² and 59 x 10⁶ N/m². Argon was used as a shielding gas to prevent oxidation of material during laser boriding.

2.5. Microstructure
After these treatments, the specimens were metallographically polished and etched using 2% Nital. Microstructures were taken at suitable magnifications using Zeiss axiovert optical microscope.

2.6. Microhardness
Vickers microhardness was measured at the surface as well as along the transverse section at 50 g load using Mitutoyo microhardness tester.

2.7. Fracture toughness
Fracture toughness (Kc) of the continuously pack borided, interrupted pack borided and multi-component (Ni, Cr and B) laser borided specimens were estimated by micro-Vickers
indentations using Vickers microhardness testers namely Mitutoyo and Zwick on polished cross-sections employing ASTM E384 standard. The fracture toughness was calculated from the following equation [3]:

$$K_c = 0.028 \left( \frac{E}{H} \right)^{1/2} \frac{P}{c^{3/2}}$$

where $K_c$ - Fracture toughness (Mpa. m$^{1/2}$); $E$ - Young’s modulus (N/m$^2$); $H$ - Microhardness (HV); $c$ - half crack length (m) and $P$ - applied load (N).

3. Results and Discussions

3.1. Optical microstructures

Figure 1 Optical microstructures of (a) continuously pack borided (b) interrupted pack borided (c) multi-component (Ni, Cr and B) laser borided specimen (treated at $E = 47 \times 10^6$ J/m$^2$) (d) multi-component (Ni, Cr and B) laser borided specimen (treated at $E = 59 \times 10^6$ J/m$^2$)

The Figure 1(a) illustrates a growth of saw tooth boride needles. It has been observed that the case depth of the continuously pack borided needles is not uniform. The case depth of the continuously pack borided needles was found in the range of 78 - 114 µm. It was reported that the continuously pack borided layer formed on the AISI 1020 steel specimen has good bonding with the steel due to acicular nature of borides [1]. Microcracks were observed in the boride layer. Grain coarsening at the core was observed due to long time exposure (3 h) at high temperature (950 °C). The case depth of the interrupted pack borided needles was found in the range of 55 – 63 µm. It has been observed from the Figure 1(b) that the interrupted pack borided needles are shorter and more uniform than continuously pack borided needles. Because of the interrupted treatment cycle, mechanical properties of the core are less disturbed as compared with continuously pack borided specimens treated at same temperature and time condition. It has been observed from Figure 1(c & d) that the interface between multi-component (Ni, Cr and B) laser borided layer and transition zone appears almost flat. It has been observed that the structure of the multi-component (Ni, Cr and B) laser borided layer was less uniform when the specimens were treated at lower energy densities. As the energy density was increased, the structure becomes more homogeneous and it contained fewer amounts of B$_4$C particles. The thickness of the multi-component laser (Ni, Cr and B) borided layer was found in the range of 210 – 220 µm.
Generally, dendritic microstructure has been observed in the multi-component (Ni, Cr and B) laser borided layer. Use of a laser beam enabled the reduction in the time formation of the boride layer as compared to the continuous pack boriding and interrupted pack boriding treatments.

3.2. Microhardness profiles
Figure 2 (a-d) illustrates microhardness profiles of continuously pack borided, interrupted pack borided and multi-component (Ni, Cr and B) laser borided (treated at $E = 47 \times 10^6 \text{ J/m}^2$ and $E = 59 \times 10^6 \text{ J/m}^2$).

The surface microhardness of continuously pack borided specimen was around $1858 \text{ HV}_{0.05}$. This high surface hardness is due to the formation of hard FeB phase. It was observed from microhardness profile Figure 2(a), there is a great scatter in the microhardness along the depth of continuously pack borided specimen. The microhardness was found to vary from $1858 \text{ HV}_{0.05}$ to $910 \text{ HV}_{0.05}$ in the case of continuously pack borided specimen. This great scatter in microhardness indicates greater inhomogeneity in the boron and carbon concentrations in the continuously pack borided layer. It was observed from Figure 2(a) that there is a sudden drop in microhardness from $910 \text{ HV}_{0.05}$ to $190 \text{ HV}_{0.05}$. When this type of borided specimen, subjected to impact/local loading during operation, catastrophic failure may happen. The surface hardness of interrupted pack borided specimen was less (around $1556 \text{ HV}_{0.05}$) as compared with continuously pack borided specimen. It was observed that hardness scatter is much lower than continuously borided specimen. The microhardness was found to vary from $1556 \text{ HV}_{0.05}$ to $1437 \text{ HV}_{0.05}$. The microhardness profile of multi-component (Ni, Cr and B) laser borided specimens treated at $E = 47 \times 10^6 \text{ J/m}^2$ is shown in the Figure 2(c). The surface hardness was found to vary between 1652-1732 HV. Smooth hardness gradient has been obtained as compared with
continuous and interrupted pack borided layers. The smooth hardness gradient may result in significant enhancement in toughness. Eventually the reduction in hardness as compared with continuously pack borided specimens can result in lower tensile strength [9]. The microhardness profile of multi-component (Ni, Cr and B) laser borided specimens treated at $E = 59 \times 10^6$ J/m$^2$ is shown in the Figure 2(d.). The surface hardness was found to vary from 1598 – 1602 HV. It has been observed that by increasing the energy density of laser beam, the surface hardness reduces significantly. However, very smooth hardness gradient was obtained.

3.3. Fracture toughness

Figure 3 (a-d) illustrates Vickers micro indentation mark with a crack for continuously pack borided, interrupted pack borided and multi-component (Ni, Cr and B) lased borided (treated at $E = 47 \times 10^6$ J/m$^2$ and $E = 59 \times 10^6$ J/m$^2$).

The loads of 1 N, 2 N and 2.9 N were used for determining fracture toughness of continuously pack borided layer. The microindentation was performed on continuously pack borided layer (at Fe$_2$B zone). Half crack lengths were tabulated. It had been reported by several authors that the young’s modulus of Fe$_2$B phase was approximately 295 GPa [1]. Fracture toughness values of continuous pack borided layer are given in Table 1.

| Load (P) (N) | Radial half crack length (C) (µm) | Fracture toughness (MPa m$^{1/2}$) |
|-------------|-----------------------------------|-----------------------------------|
| 1           | No Cracks                         | -                                 |
| 2           | 20.3                              | 2.51                              |
| 2           | 19.6                              | 2.71                              |
| 2.9         | 20.9                              | 3.43                              |
The fracture toughness of continuously pack borided layer was found in the range of 2.51 – 3.43 MPa.m\(^{1/2}\).

The loads of 1 N, 2 N and 2.9 N were used for determining fracture toughness of interrupted borided layers. The microindentation was performed on interrupted borided layer (on Fe\(_2\)B layer). Half crack lengths were tabulated. Fracture toughness values of interrupted boride layer are given in the Table 2.

**Table 2 Results of fracture toughness test of interrupted pack borided specimens**

| Load (P) (N) | Radial half crack length (C) (µm) | Fracture toughness (MPa.m\(^{1/2}\)) |
|--------------|----------------------------------|-------------------------------------|
| 1            | No crack                         | -                                   |
| 2            | 18.1                             | 3.49                                |
| 2            | 18.3                             | 3.44                                |
| 2.9          | 19.4                             | 4.72                                |
| 2.9          | 18.9                             | 4.91                                |

The fracture toughness of interrupted boride layer was found in the range of 3.44 – 4.91 MPa. m\(^{1/2}\). An improvement in fracture toughness as compared to the continuously pack borided layer has been observed. It has been concluded from the experimental results that interrupted pack boride layer are relatively ductile as compared with continuously borided layer. This may be due to more uniform nature of interrupted borided layer as compared with continuously pack borided layer.

The loads in the range of 39.2 N to 68.6 N were used for determining fracture toughness of multi-component (Ni, Cr and B) laser borided layers. Unlike continuously pack borided and interrupted pack borided layers, multi-component (Ni, Cr and B) laser borided layers have different phases that are mixed together. The various phases are: Fe\(_2\)B, FeB, NiB, Ni\(_4\)B\(_3\), CrB, Cr\(_2\)3C\(_2\) and Fe\(_7\)C\(_3\). The wt.% and vol.% of each phase in the boride layer were determined from XRD data. The young’s modulus and density of individual phases are well known. Hence, the young’s modulus and density of composite boride layer were determined using the principle of rule of mixtures.

The young’s modulus was calculated as 298 GPa approximately for a specimen treated at E = 47 x 10\(^6\) J/m\(^2\). No cracks were observed when the applied load is below 49 N. Half crack lengths were tabulated. Fracture toughness values of multi-component (Ni,Cr and B) laser borided layer developed at E = 47 x 10\(^6\) J/m\(^2\) are given in the Table 3.

**Table 3 Results of fracture toughness test of multi-component (Ni, Cr and B) laser borided specimens treated at E = 47 x 10\(^6\) J/m\(^2\)**

| Load (P) (N) | Radial half crack length (C) (µm) | Fracture toughness (MPa.m\(^{1/2}\)) |
|--------------|----------------------------------|-------------------------------------|
| 49           | No cracks                         | -                                   |
| 68.6         | 75.4                             | 13.8                                |

The young’s modulus was calculated as 290 GPa approximately for a specimen treated at E = 47 x 10\(^6\) J/m\(^2\). Half crack lengths were tabulated. Fracture toughness values of multi-component (Ni, Cr and B) laser borided layer developed at E = 59 x 10\(^6\) J/m\(^2\) are given in the Table 4.
Table 4 Results of fracture toughness test of multi-component (Ni, Cr and B) laser borided specimens treated at $E = 59 \times 10^6 \text{ J/m}^2$

| Load (P) (N) | Radial half crack length (C) (µm) | Fracture toughness (MPa. m$^{1/2}$) |
|--------------|-----------------------------------|-------------------------------------|
| 39.2         | No cracks                         | -                                   |
| 49           | 50.1                               | 18.3                                |

A significant improvement in fracture toughness has been observed from the experimental results. This may be due to (i) better distribution of boron and other alloying elements in the boride layer due to laser treatment and (ii) relatively more uniform boride layer (less degree of inhomogeneity) as compared with continuously pack borided layer.

Conclusions
From the fracture toughness ($K_c$) measurements the following conclusions have been made:

(i) It has been concluded that cracks observed at high loads in the case of multi-component (Ni, Cr and B) laser borided layers as compared with continuously pack borided and interrupted pack borided layers.

(ii) Multi-component (Ni, Cr and B) boride layer is more ductile as compared with continuously pack borided and interrupted pack borided layers.

(iii) For multi-component (Ni, Cr and B) laser borided layers developed at higher energy densities (above $59 \times 10^6 \text{ J/m}^2$) cracks were not observed even at 68.6 N load. However, significant drop in surface hardness has been observed.

References
[1] Kulka M et al 2015 Opt. Laser Eng. 67 163
[2] Yusuf Kayali and Sukru Taktak 2015 J. Adhes. Sci. Technol. 29 2065
[3] Genel K et al 2003 Mat. Sci. Eng. A 347 311
[4] Uslu I et al 2007 Mater. & Design 28 55
[5] Dybkov V I et al 2006 J.Mater. Sci. 41 4948
[6] Prince M et al 2011 High Temp. Mater. Pr. 29 313
[7] Gopalakrishnan P et al 2002 Metall. Mater. Trans. A 33A 1475
[8] Gopalakrishnan P et al 2001 Scripta Mater. 44 707
[9] Xu C H et al 2001 J. Mater. Process. Tech. 108 349
[10] Davis J A et al 1998 Surf. Coat. Tech. 103-104 52
[11] Novakova A A et al 2004 J. Alloy. Compd. 383 108
[12] Bourithis L and Papadimitriou G D 2009 Wear 266 1155
[13] Prince M 2012 Some studies on laser boriding of nickel and chromium electroplated AISI 1020 steels Ph.D Thesis Anna Univerity Chennai India