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Surface melting of a medium carbon steel by laser treatment

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A 0.4 wt % C normalized carbon steel has been graphite-coated and irradiated in air with a cw-CO_2 laser beam at 1.3 kJ cm^{-2} to melt a near-surface layer of material. The solidified and the solid-state transformed regions have been investigated by means of surface Mössbauer spectroscopy, X-ray diffraction, metallography and microhardness measurements. Nature and morphology of the phases have been determined in each region, and their origin discussed in relation to the conditions of the treatment.

1. Introduction

Laser beams provide a controlled source of heat by which materials can be machined or surface treated with exceptionally high rates of heating and cooling. Moreover, highly localized treatments, hardening of components complex in shape and size and a reduction in post heat-treatment machining operations are allowed. In the technology of surface treating iron alloys, in particular, new processing methods have been developed using continuous-wave CO_2 lasers emitting up to 15 kW or more. Examples of components for the automotive and aircraft industries which are reported in the literature to have been hardened by means of laser induced solid-state martensitic transformations include gear teeth, crankshafts, journals, cylinder bores and piston rings [1–3].

In the present work, a laser treatment has been considered involving the near-surface melting of a medium carbon steel, for the following reasons: controlled surface melting and high speed solidification of iron alloys is currently utilized to obtain structures and properties of practical interest. For example, cast iron camshafts have been laser melted to a depth of 1 mm and increases in surface hardness, wear and corrosion resistance have been claimed for the treatment [4]. The effects of a surface melting followed by self-quenching on the properties of an M2 tool steel and a Cr-rich 440-C alloy have been studied by Strutt *et al.* [5]: the laser-melted zone of M2 contained δ-ferrite, austenite and residual amounts of M_2C and $M_{23}C_6$ carbides, while the laser melting produced a δ-ferrite/Cr_7C_3 carbide eutectic structure in the 440-C alloy. Secondly, it is possible that localized surface melting may unwantedly occur when solid-state surface hardening as a consequence of some non-uniform heat absorption by the irradiated alloy occurs.

2. Experimental details

Plates 10 mm thick of a normalized 0.4 wt% C carbon steel have been coated with an anti-reflective film of colloidal graphite and then irradiated in air by a cw-CO_2 laser beam, with an incident power of 10 kW, at a translation speed of 4.5 m min^{-1}. A focused beam, arising from a 15 kW AVCO source, was oscillated to produce a 10 mm × 10 mm spot uniform in specific power. The beam–material interaction time was 0.13 sec, and the energy density 1.3 kJ cm^{-2}. The graphite coatings have been adopted by considering the detrimental effects that the more used anti-reflection phosphating treatments exert on the mechanical properties of the laser-irradiated iron alloys.

Cross-sections of some of the treated samples were cut and metallographically prepared for examination by abrading on SiC paper and final polishing with diamond paste; during each preparation stage wet procedures were employed to avoid undesirable heating effects. The sections were chemically etched in a 2% nital reagent. A Reichert MeF optical microscope was utilized for metallography. The microhardness measurements were carried out using a Vickers indenter with a 0.1 N and, occasionally, a 0.5 N load. For fractographic analysis, some laser-treated specimens have been V-notched, fractured by impact loading and then observed under a Jeol JSM-P15 scanning electron microscope. X-ray diffraction analyses have been carried out using a Philips PW 1720 X-ray generator, equipped with a computer-controlled goniometer, and the Co$K\alpha$ radiation.

The surface Mössbauer measurements were performed using the experimental apparatus described by Carbucicchio [6] which detected the K X-rays (6.4 keV) and the K-shell conversion electrons (7.3 keV) resonantly re-emitted by the ^{57}Fe atoms. As far as the electrons are concerned, regions of different depth have been sampled by selecting electrons emerging from the surface with energies in the 5.5 to 7.3, 6 to 7.3 and 6.5 to 7.3 keV ranges. The thickness of the analysed surface layers was estimated following the method of Krakowski and Miller [7], with a correction for the escape of detected radiation for different phases [8]. A 40mCi ^{57}Co(Pd) source has been used. The spectra were measured at room temperature and computer-fitted to a set of Lorentzian peaks. In the spectra shown, the isomer shifts were normalized with respect to the pure iron.

3. Results and discussion

Fig. 1 shows a metallographic cross-section of the laser-treated 0.4 wt% C steel. The outermost region assumed a dendritic, bi-phase structure as a result of surface melting and subsequent solidification processes. The thickness of this region extended down to about 20 μm. Locally, at the largest laser-produced steep drops, thicknesses up to about 80 μm have been measured. The hardness values measured in the solidified region were varied significantly, with maxima around 9 kN mm^{-2}.

Surface melting also influenced the morphology of the solid-state transformed zone. Under the solidified region, Fig. 1 shows a white layer about 5 to 10 μm thick and an inner zone consisting of heterogeneous martensite. The thermally affected zone extended down to about 0.8 mm below the surface. At a higher magnification, the white layer displays a three-phase structure, i.e. (i) a large-grained matrix, the grain being generally oriented normal to the external surface, (ii) a darker phase which segregated at grain boundaries of the matrix (Fig. 2), and (iii) acicular

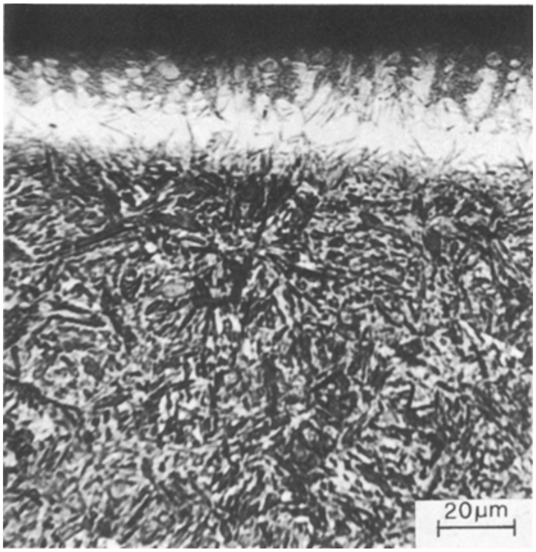

Figure 1 Optical micrograph of a laser-treated and cross-sectioned 0.4 wt% C carbon steel, showing an outer bi-phase solidified region and, in the solid-state transformed region, a white layer and a zone of heterogeneous martensite.

Figure 2 Tapered section of the white layer shown in Fig. 1: a darker phase segregated at grain boundaries of the matrix.

Figure 3 As in Fig. 2, showing coarse and acicular crystals of martensite which appear having been nucleated and grown starting from grain boundaries of the matrix.

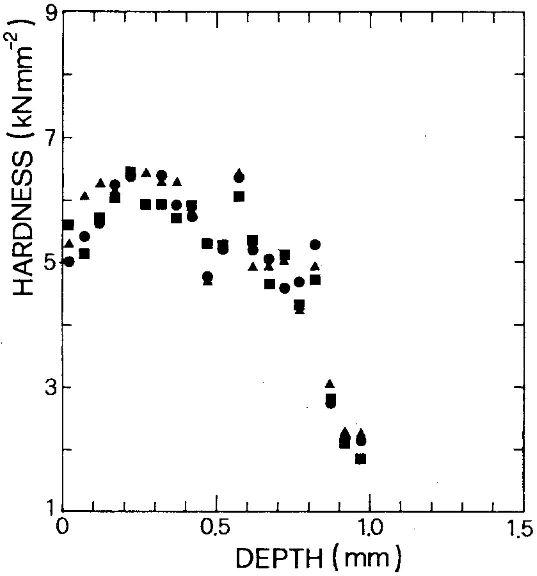

Figure 4 Three hardness profiles measured through the laser-transformed regions, normal to the external surface and about 0.3 mm close to each other (Vickers indenter, 1 N load).

and coarse crystals of martensite, which seemingly nucleated and grew starting from the same boundaries (Fig. 3). This layer, moreover, is appreciably softer than the adjacent regions: the measured hardness values are around 6 kN mm^{-2}.

The hardness profiles of the martensite zones which are shown in Fig. 4 exhibited large fluctuations, nevertheless, a maximum of about 6.5 kN mm^{-2} could be observed at 0.2 to 0.3 mm from the external surface. At increasing depths, a series of structures progressively prevails, resulting from partial transformation of the original ferritic-pearlitic matrix. Correspondingly, hardness decreases to values typical of the unaffected alloy.

Fig. 5a shows the 6.4 keV X-ray Mössbauer spectrum (estimated depth, about 20 μm) measured for the 0.4 wt % C steel before the laser treatment. This spectrum can be decomposed into a major component due to iron atoms in environments as a pure bcc iron, and to minor ferromagnetic components due to those atoms perturbed by alloying elements and impurities present in the steel. The small amount of cementite in the sample does not significantly contribute to the spectrum.

Fig. 5b shows the corresponding 6.4 keV X-ray Mössbauer spectrum for the laser-treated 0.4 wt % C steel. It can be described in terms of three superimposed spectra:

1. a high intensity central peak and a quadrupole doublet, due to paramagnetic austenite [9, 10];
2. a low intensity six-peak spectrum, whose Mössbauer parameters agree with those reported in the literature for cementite [9, 11];
3. a multi-component ferromagnetic spectrum, which can be attributed to martensite. As is well known [9–11], the Mössbauer spectrum of martensite consists of several contributions arising from the first, second, third to fourth and further neighbour iron atoms of each carbon atom located in one of the octahedral sites of the structure along the z-axis. In the present case, owing to the low intensity of the spectrum, only the components corresponding to iron atoms in high-populated sites are detectable.

In agreement, the X-ray diffraction pattern of the same laser-treated specimen shows reflections which can be attributed to cementite, austenite and martensite (Fig. 6).

In order to obtain information undoubtedly arising from the solidified region and, in particular, from very thin surface layers, conversion electron Mössbauer spectra have been measured for the same sample as Fig. 5b. Fig. 5c show the spectrum measured by detecting the conversion electrons in the 6.5 to 7.3 keV energy range (estimated

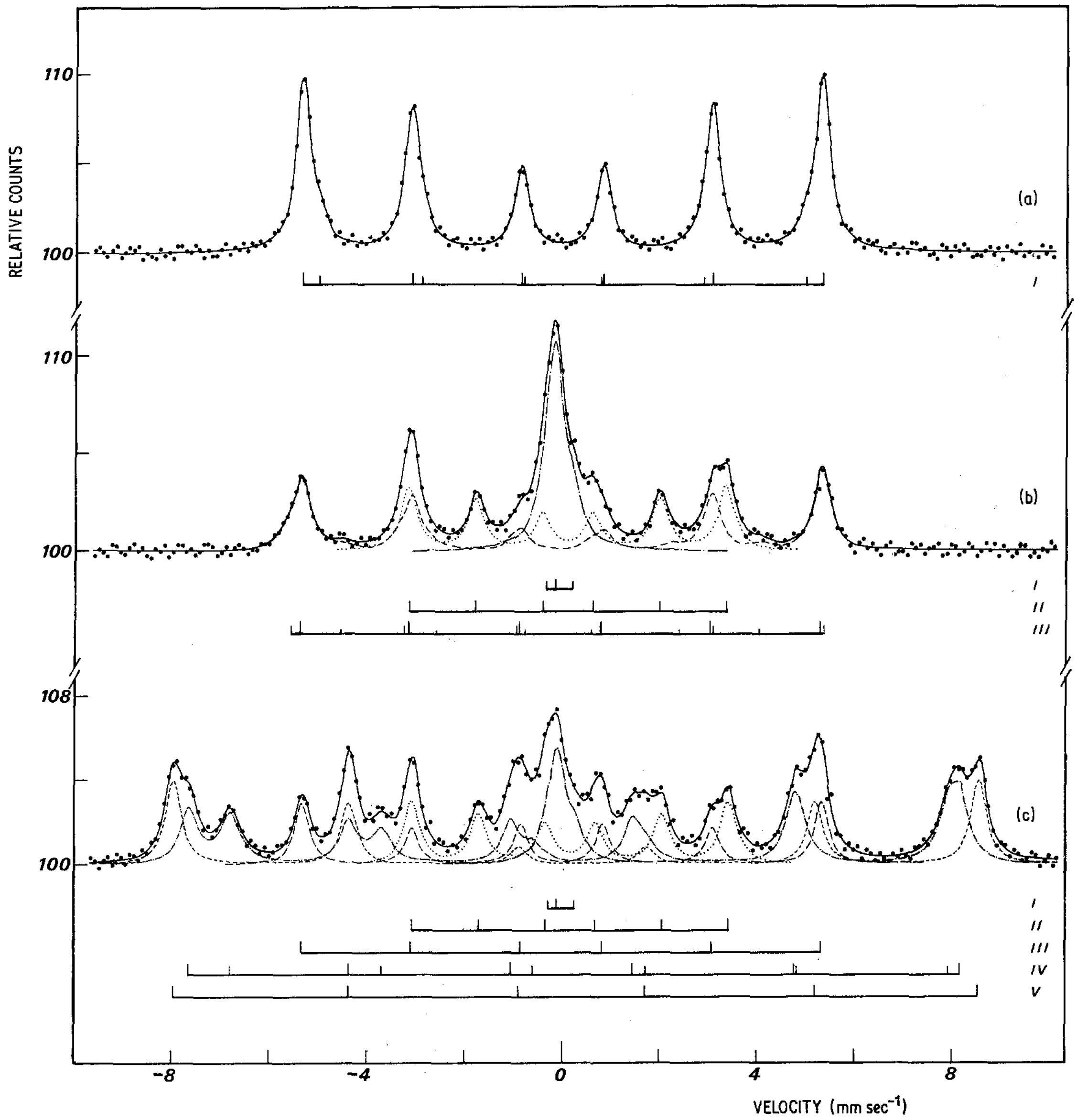

Figure 5 Room temperature surface Mössbauer spectra for: (a) untreated 0.4 wt% C steel (6.4 keV X-rays), and (b) laser-treated steel (6.4 keV X-rays), (c) laser-treated steel (6.5 to 7.3 keV conversion electrons).

thickness of the analysed surface layer, about 100 nm). It can be interpreted as the superposition of several spectra:

I. a central peak and a quadrupole doublet, due to paramagnetic austenite;

II. a six-line spectrum, due to cementite;

III. another sextet, due to iron atoms in environments as in pure bcc iron: taking into account the relatively low cooling rates of the outermost parts of the melted region, a partial austenite–ferrite transformation can be admitted and this contribution attributed to ferrite;

IV. a broad ferromagnetic spectrum, which can be described in terms of two superimposed contributions due to iron atoms in the Fe_3O_4 compound [12];

V. a six-line spectrum, which can be attributed to iron in Fe_2O_3 oxide [13].

From a comparison between surface Mössbauer measurements and metallographic observations, one is allowed to conclude that the solidified bi-phase region contains dendritic austenite, presumably supersaturated in carbon, and cementite. Iron oxides are confined on the

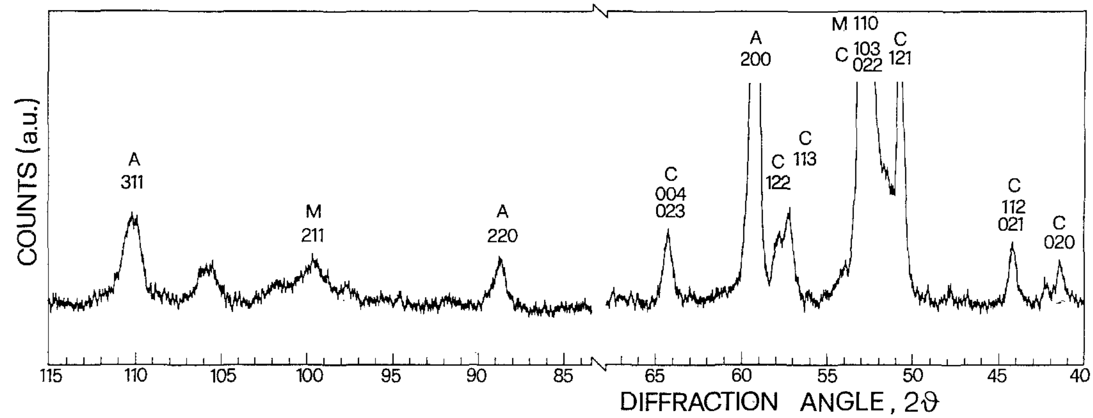

Figure 6 X-ray diffraction pattern of the laser-treated 0.4 wt % C steel (Co$K\alpha$ radiation). A, C and M mean austenite, cementite and martensite, respectively.

external surface of this solidified region, as shown by depth selective electron Mössbauer spectra. Their growth occurred despite the protective action that the anti-reflection graphite coating exerted against the oxidizing atmosphere.

In the solid-state transformed region, the comparison between spectroscopic and metallographic analyses demonstrates that the main component of the white and relatively soft layer observed over the martensite zone (Fig. 1) can only be austenite. As for the darker intergranular phase (Fig. 2), it can be reasonably identified as cementite having been separated at the grain boundaries of a carbon saturated austenite. As shown by fractographic analysis (Fig. 7), this phase constitutes a brittle network. Owing to local differences in cooling conditions, nucleation and growth of acicular crystals of martensite only were allowed at preferred sites in this layer.

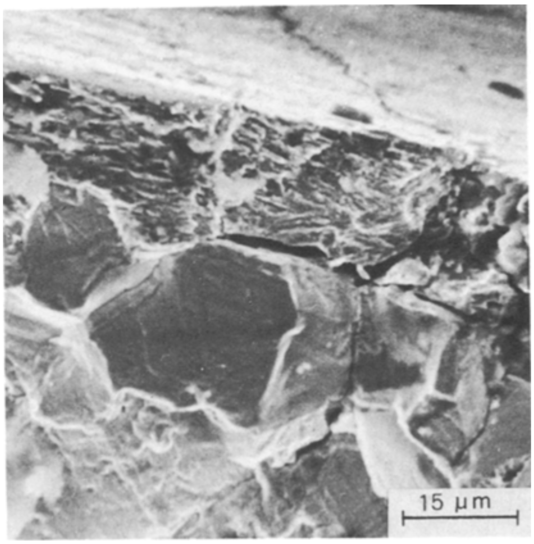

Figure 7 SEM fractograph of the laser-treated 0.4 wt % C carbon steel, showing a fully intergranular fracture of the solid-state transformed austenitic layer (the white layer in Fig. 1).

4. Conclusions

The 0.4 wt % carbon steel, treated at 1.3 kJ cm^{-2}, shows an outer solidifed region and an inner solid-state transformed region. Analysing the first, it can be concluded that a heterogeneous absorption of energy occurred. The uniformity of energy distribution in the incident beam being carefully controlled, this behaviour must be attributed to irregular burning in air of the anti-reflection graphite coating, formation of plasmas, etc. In any case, the melting temperature was reached at the more absorbing surface zones and graphite dissolved in the melt causing further liquation. The cooling of this carbon-enriched melted alloy allowed an austenite–cementite structure to be formed, whose hardness fluctuated reaching a maximum of about 9 kN mm^{-2}. Growth of iron oxides occurred at the external surface of the solidified region, in spite of the presence of a graphite coating.

In the solid-state transformed region, a first layer of large-grained austenite formed, containing intergranular cementite and isolated, coarse, acicular crystals of martensite, whose hardness fluctuated around an average value of 6 kN mm^{-2}. The use of graphite as anti-reflection material and the re-heating capacity of the upper melted material appear to have largely controlled these transformations.

The main part of the solid-state transformed region contains martensite, whose heterogeneity and coarseness lead to large fluctuations and inadequacy in hardness values. In this zone, the increase of hardness with depth can be related to decreasing amounts of retained austenite. This conclusion is strongly supported by the presence, upon the martensitic zone, of a mainly austenitic layer.

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