

Structural analysis of electron beam irradiated steel/molybdenum systems

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Layered systems, obtained by e-beam irradiation of molybdenum sheets mechanically clamped to ARMCO iron, plain carbon steel and high alloy austenitic steel substrates, have been analysed by means of conversion X-ray Mössbauer scattering and X-ray diffraction. When the substrate is iron or carbon steel, the structure of the surface layer is completely ferritic with a certain amount of iron-atoms coordinated with molybdenum atoms. If the substrate is fully austenitic, the e-beam treated surface structure presents a broad ferritic-martensitic component. The influence of the cooling rate of the e-beam molten layer is also discussed in relation to the observed surface structures.

1. Introduction

Electron beam cladding and alloying are today acknowledged as advanced treatments able to modify the surface composition, microstructure and properties of materials. The possibility of improving the corrosion resistance of steels has recently been demonstrated by the irradiation of bilayers formed by a substrate of plain carbon steel and an overlayer of AISI 316 stainless steel [1, 2]. The sheet of stainless steel was mechanically clamped to the substrate and then irradiated with the electron beam. It is important to point out that the operating conditions of this treatment technique can be controlled in order to obtain a prefixed melt depth. Then, controlled amounts of the substrate are remixed together with the overlayer, so that the resulting

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chemical composition and microstructure of the molten zone depends on the dilution of the alloy elements of the overlayer material [3]. Also, electron beam alloying of molybdenum with AISI 304 as a substrate has been investigated. The surface enrichment with this element has been shown to improve the corrosion behavior of the AISI 304 stainless steel substrate [4]. The purpose of this work is to investigate by X-ray diffraction (XRD) and conversion X-ray Mössbauer spectroscopy (CXMS) the microstructure obtained by alloying a thin sheet of molybdenum on substrates of pure iron, plain carbon steel and fully austenitic stainless steel.

2. Experimental

Sheets of ARMCO iron, C40 plain carbon steel (about 0.35 wt.% C), and AISI 304 austenitic stainless steel (nominal composition in wt.%: Cr = 18, Ni = 10, C + N \leq 0.07) having dimensions 300 \times 30 \times 5 mm were used as a substrate. Sheets of molybdenum 0.25 mm thick were mechanically clamped on the substrate. The bilayers molybdenum/substrate were then irradiated by a high power electron beam with the following parameters: accelerating voltage of 50 kV, beam currents equal to 70, 74 and 90 mA. A rectangular electron spot of 25 \times 2 mm was scanned over the surface with a frequency of 500 Hz and translated along the shorter dimension with a travel speed of 0.5 m/min. The width of the treated zone was then 25 mm. The electron spot was scanned three times over the surface to homogenize the composition of the alloyed zone.

Cross sections of the treated samples were polished and etched according to the usual metallographic procedures, and observed by light microscopy. The treated surfaces were analysed by XRD and CXMS before and after mechanical removal of the outer rough surface layer (0.3–0.5 mm). XRD was performed in the Bragg–Brentano geometry using Mo-K α radiation. CXM spectra were recorded with a standard spectrometer using a ^{57}Co :Rh source and a flowing gas (90% argon, 10% CH $_4$) proportional detector.

3. Results and discussion

Observation of the sample cross sections indicates the formation of a molten layer of rather uniform thickness that has been estimated to be around 2 mm. The dilution of molybdenum into the melted zone then resulted to be 10–15% in weight.

The XRD patterns of the e-beam alloyed sample were obtained after mechanical removal of the outer rough layer. In the case of ARMCO iron and plain carbon steel substrate, the XRD patterns seem to be identical and were indexed as bcc α -Fe with $a_0 = 0.2865 \pm 0.0005$ nm. A slight increase in the lattice parameter of α -Fe, which could originate from the incorporation of a few percent molybdenum atoms [5], was not detected under our experimental conditions. The XRD pattern of the sample with the AISI 304/substrate shows, after the e-beam treatment, the presence of an additional α -Fe broad peak with an intensity rather lower than that of austenite. A

rough evaluation of the composition of the molten layer gives the following values in wt.%: Cr = 15, Ni = 9, Mo = 15, (C + N) = 0.07. We can use these values to obtain the chromium and nickel equivalents, and enter the Schaeffler diagram [6] to confirm that the final structure should indeed be a mixture of austenite and ferrite.

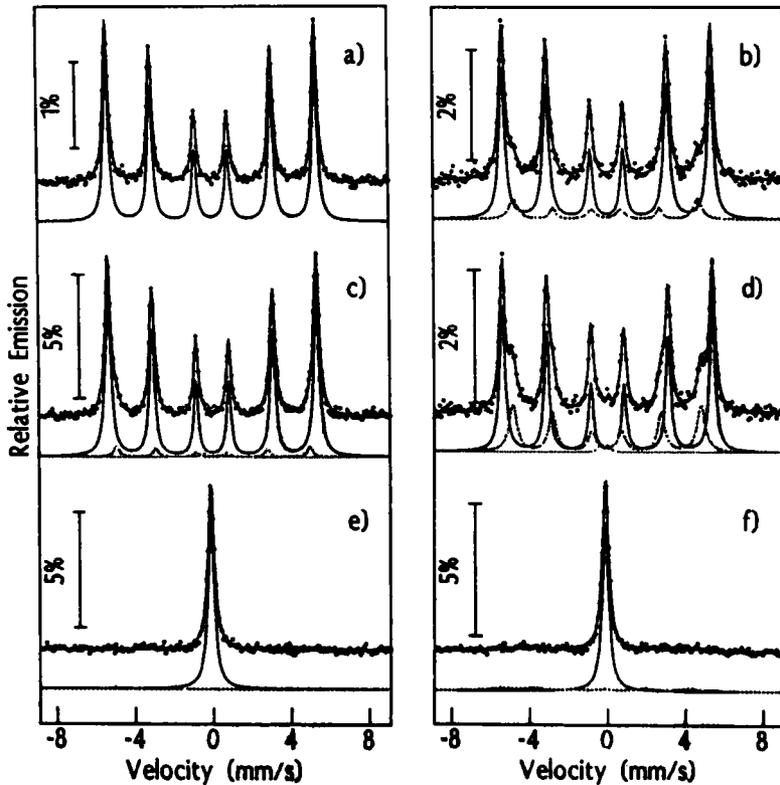


Fig. 1. Conversion X-ray Mössbauer spectra of Mo/X before and after e-beam irradiation: X = ARMO iron (a), (b); C40 steel (c), (d); AISI 304 steel (e), (f), respectively.

The CXM spectra of fig. 1 have been quantitatively interpreted according to table 1. It is worthwhile to remark that the CXM spectra did not show any substantial difference before and after mechanical removal of the outer rough layer, which demonstrates that the molten zone does not have any structural gradient in depth. This is a consequence of the multiple pass and of the effective mixing due to the e-beam treatment.

The CXM spectrum of ARMCO iron after irradiation displays, besides the characteristic magnetic spectrum of α -iron at 33 T, an additional magnetic pattern, at about 30 T, due to iron atoms with one or more molybdenum atoms nearest

Table 1

Fitted parameters of CXM spectra for Mo/X (X = ARMCO iron, C40 steel and AISI steel) interfaces before and after electron beam irradiation.

X =	ARMCO Fe/Mo		C40 steel/Mo		AISI 304 steel/Mo	
	before irradiation	after irradiation	before irradiation	after irradiation	before irradiation	after irradiation
<i>W</i>	0.29	0.23	0.27	0.29		
<i>B</i>	33.15	33.20	33.11	33.35		
<i>IS</i>	0.00	0.01	0.00	0.01		
<i>QS</i>	0.00	0.00	0.00	-0.01		
<i>I</i>	100.0	84.1	92.7	67.8		
<i>W</i>		0.44	0.31	0.43		1.60
<i>B</i>		29.77	30.73	29.78		30.00
<i>IS</i>		0.02	-0.01	0.00		0.00
<i>QS</i>		-0.05	0.02	0.03		0.20
<i>I</i>		15.9	6.0	31.5		18.8
<i>W</i>			0.26	0.30		
<i>IS</i>			0.00	0.07		
<i>QS</i>			0.40	0.40		
<i>I</i>			1.3	0.7		
<i>W</i>					0.39	0.37
<i>IS</i>					-0.12	-0.10
<i>I</i>					100.0	81.2

IS, *QS*, *W* (linewidth) in mm/s; *B* in T; *I* (relative area) in %; *IS* values referred to metallic α -iron.

neighbors. The computed relative percent area of this component agrees with the estimated molybdenum dilution in the irradiated samples in which the Mo/Fe atomic ratio is about 0.1.

The case of plain carbon steel is a little more complicated. The spectrum of this sample has two additional patterns, both before and after irradiation; a magnetic one at about 30 T and a small quadrupole doublet. The doublet is due to iron atoms in a non-magnetic carbide phase and contributes more or less in the same way to both spectra. The second magnetic component is due to iron atoms in the α -Fe phase having carbon and molybdenum atom nearest neighbors. The difference in the relative intensity of this spectral component before and after irradiation is nearly twice the corresponding component in the irradiated ARMCO iron sample. Thus, the presence of carbon atoms in solid solution before irradiation enhanced this component in some way. It is to be pointed out, however, that also the rapid cooling after irradiation can contribute strongly to this spectral component due to martensitic transformation.

The irradiated austenitic steel gives rise to a spectrum with an additional broad magnetic component which is likely attributed to an α -bcc phase originated by the rapid cooling. The qualitatively agrees with the XRD data previously discussed. From a quantitative point of view, the structure evaluation on the basis of the Schaeffler diagram (about 40% ferrite and 60% austenite) and of Mössbauer subspectral intensities shown in table 1 could be in better agreement with a more accurate evaluation of the molten layer thickness by microanalytical investigation on a wide set of treated samples.

4. Conclusions

With this work, we have shown once again that Mössbauer spectroscopy, combined with X-ray diffraction, is of great utility in the study of the metallurgical features obtained with the rather new surface treatment method based on e-beam irradiation.

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