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Effects of focused ion beam milling on austenite stability in ferrous alloys

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ABSTRACT

The susceptibility of fcc austenite to transform to bcc during focused ion beam milling was studied in three commercial stainless steels. The alloys investigated, in order of increasing austenite stability, were: (i) a model maraging steel, Sandvik 1RK91; (ii) an AISI 304 austenitic stainless steel; and (iii) AL-6XN, a super-austenitic stainless steel. Small trenches were milled across multiple austenite grains in each alloy using a 30 kV Ga^+ ion beam at normal incidence to the specimen surface. The ion beam dose was controlled by varying the trench depth and the beam current. The factors influencing the transformation of fcc austenite to bcc (listed in order of decreasing influence) were found to be: (i) alloy composition (i.e., austenite stability), (ii) ion beam dose (or trench depth), and (iii) crystallographic orientation of the austenite grains. The ion beam current had a negligible influence on the FIB-induced transformation of austenite in these alloys.

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1. Introduction

Focused ion beam (FIB) milling has emerged as an effective specimen preparation tool in materials science. In a FIB instrument, a finely focused ion beam from a liquid metal ion source (usually Ga^+) impacts the specimen, leading to localized sputtering that can be controlled to mill nanometer-scale features. A dual beam FIB instrument, which also contains an electron source, can additionally be equipped with an electron backscatter diffraction (EBSD) detector for crystallographic orientation analysis. This combination enables the dual beam FIB to acquire crystallographic information in three dimensions by sequentially milling the sample with the FIB and analyzing each freshly produced surface by EBSD [1–6].

Irradiating a solid with a beam of energetic ions, however, invariably results in some degree of microscopic disruption and heat generation. The various interactions that can occur between the ion beam and the specimen have been reviewed by several authors (e.g., see [7–13]). In crystalline materials, ion beam irradiation can generate extensive lattice defects which, for a sufficient implantation dose, can lead to phase transformations and/or complete amorphization in the material.

The present study investigates the stability of austenite in three steels as a function of several FIB milling conditions. The three steels examined represent various levels of inherent austenite stability, based on their alloy content. The austenite stability was evaluated in each alloy as a function of ion dose and beam current to determine the relative response to those milling parameters. This study was undertaken in large part to provide a critical knowledge base for potential FIB-based three-dimensional analytical experiments in ferrous alloys containing significant volume fractions of austenite, but it is also relevant to the FIB preparation of transmission electron microscopy specimens. Use of such FIB milling techniques for 3D microstructural studies has been reported previously in many non-ferrous alloy systems (e.g., see [1,4–6,14]) but less frequently in ferrous alloys (e.g., [1,2,15]).

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2. Experimental Procedures

Three commercial stainless steels were investigated in this study: (i) a model maraging steel, Sandvik 1RK91, which forms isothermal martensite starting at temperatures around 40 °C and achieves a maximum transformation rate at −40 °C [16]; (ii) an AISI 304 metastable austenitic stainless steel; and (iii) an AL-6XN super-austenitic stainless steel. These three alloys were chosen to represent varying degrees of inherent austenite stability based on their increasing levels of alloying elements. Published alloy compositions are provided in Table 1.

Metallographic preparation of the specimens was carried out using conventional techniques. Final polishing of the surface was accomplished with a solution of 20% hydrogen peroxide (30%) and 80% colloidal silica solution. The specimens were examined in an FEI Nova 600 NanoLab DualBeam™ SEM/FIB equipped with an HKL Channel 5 EBSD system. Some EBSD analyses were also performed with a JEOL JSM-7001F SEM, equipped with an EDAX-TSL Hikari EBSD detector. Rectangular trenches (usually ~$5\times5\ \mu m^2$) were milled using a 30 kV Ga^+ beam at normal incidence to the specimen surface to assess the effect of ion beam irradiation on the austenite stability. While most practical milling situations (e.g., in serial sectioning or in transmission electron microscopy specimen preparation) utilize high glancing angles of incident radiation, normal incidence was used in this study to provide a more reproducible ion dose and to confine the resulting damage to a well-defined region suitable for subsequent EBSD analysis. The sputtering yield is significantly less for incident radiation as compared with high glancing angles, but the relative damage induced by the beam is comparable [7,9].

The trenches were based on preprogrammed raster patterns in the FEI control software, which are defined by a number of parameters. The pattern depth and beam current were varied to adjust the delivered dose of the ion beam. The depth of the milled pattern (a nominal value based on the sputtering yield of silicon) is directly proportional to the delivered dose. The beam current (the rate of delivery of ions to the specimen) was varied from 0.1 to 5.0 nA. The dwell time (the duration that the beam spends at each step) and the overlap (the distance between adjacent steps) were held constant at 1 µs and 50% overlap, respectively. The resulting damage and austenite transformation were examined using electron forescatter detector orientation contrast imaging and EBSD.

3. Results and Discussion

A typical forescatter electron image of the polished surface of the Sandvik 1RK91 alloy is shown in Fig. 1(a). EBSD analyses of this and other regions indicated that the initial microstructure was primarily martensitic, with a minor constituent of small (~ 3 µm) equiaxed austenite grains. The surface morphology of the martensite and austenite constituents appeared similar in secondary electron images, although forescatter electron images typically displayed more visible striations in the martensitic material in the Sandvik 1RK91 alloy. Three austenitic grains are contained within the indicated boxed area, as revealed (in red) in the superimposed EBSD phase map in Fig. 1(b).

Because the volume fraction of austenite was relatively small, a series of narrow trenches, 0.5 µm wide and 10 µm long, was milled across the austenite grains. Milling was performed with a 1.0 nA beam current of 30 kV Ga^+ ions at normal incidence to the specimen surface with an ion dose of $2.1\times10^{17}\ Ga^+\ ions/cm^2$. Fig. 1(a) shows a trench milled through one of the austenite grains. An EBSD phase map of this region, Fig. 1(b), indicates that the fcc (red) austenite in that milled region had transformed to bcc (blue). Fig. 1(c) shows the result of milling a second trench through a different austenite grain. A comparison of the superimposed phase identification maps in Fig. 1(b) and (c) shows unambiguously that the FIB beam has effected a transformation from fcc austenite to bcc in the milled region.

The milling-induced decomposition of fcc austenite to a bcc phase in the Sandvik 1RK91 alloy led to a parametric study of the factors influencing such austenite decomposition. Two additional austenitic alloys with increasing levels of austenite stability (as indicated by the alloying constituents, Table 1) were selected for this study, in large part because of the marginal austenite stability of the Sandvik steel, which partially transforms to isothermal martensite at room temperature [16]. The current study thus focused predominantly on AISI 304 stainless steel due to its enhanced austenite stability as well as its widespread use and availability. An AL-6XN super-austenitic steel was also examined to evaluate the susceptibility to milling-induced transformation in an alloy with an even higher inherent austenite stability.

To evaluate the effects of ion beam dose on the stability of austenite, a series of $5\times5\ \mu m^2$ trenches was milled in the AISI 304 stainless steel using a beam current of 0.1 nA. The Ga^+ ion dose, calculated from the beam current and required milling time, was varied from 4.2 to $33\times10^{15}\ Ga^+\ ions/cm^2$, by varying the pattern depth. The forescatter electron micrograph in Fig. 2(a) shows the expected increase in trench depth with increasing ion dose. After milling, the microstructure was analyzed by EBSD, as shown in Fig. 2(b) and (c). The original microstructure was entirely austenitic and remained untransformed for doses less than $13\times10^{15}\ Ga^+\ ions/cm^2$. For moderate doses (13 to $25\times10^{15}\ Ga^+\ ions/cm^2$), the transformation was nonuniform across the milled area. For the most severe milling conditions (doses greater than $29\times10^{15}\ Ga^+\ ions/cm^2$), all of the milled area was transformed to bcc.

Table 1 – Compositions (wt.%) of alloys investigated [16–18].

	Ni	Cr	Mo	Mn	Si	Ti	Al	N	Cu	C	P	S
Sandvik 1RK91	9	12	4	0.3	0.15	0.9	0.3		2	<0.01		
AISI 304	8–10.5	18–20		2	1					0.08	0.045	0.03
AL-6XN	24	20.5	6.2	0.4	0.4			0.22	0.2	0.02	0.02	0.001

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