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Nature of gallium focused ion beam induced phase transformation in 316L austenitic stainless steel

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ABSTRACT

The microstructural evolution and chemistry of the ferrite phase (α), which transforms from the parent austenite phase (γ) of 316L stainless steel during gallium (Ga) ion beam implantation in Focused Ion Beam (FIB) instrument was systematically studied as a function of Ga^+ ion dose and γ grain orientations. The propensity for initiation of $\gamma \rightarrow \alpha$ phase transformation was observed to be strongly dependent on the orientation of the γ grain with respect to the ion beam direction and correlates well with the ion channelling differences in the γ orientations studied. Several α variants formed within a single γ orientation and the sputtering rate of the material, after the $\gamma \rightarrow \alpha$ transformation, is governed by the orientation of α variants. With increased ion dose, there is an evolution of orientation of the α variants towards a variant of higher Ga^+ channelling. Unique topographical features were observed within each specific γ orientation that can be attributed to the orientation of defects formed during the ion implantation. In most cases, γ and α were related by either Kurdjumov-Sachs (KS) or Nishiyama-Wassermann (NW) orientation relationship (OR) while in few, no known OR's were identified. While our results are consistent with gallium enrichment being the cause for the $\gamma \rightarrow \alpha$ phase transformation, some observations also suggest that the strain associated with the presence of gallium atoms in the lattice has a far field stress effect that promotes the phase transformation ahead of gallium penetration.

1. Introduction

Focussed ion beam (FIB) instruments are widely used for the preparation of samples for microstructural characterization as well as for various micro mechanical tests [1]. However, it has been observed in some materials that this technique, though widely accepted and in regular use, does introduce artefacts during sample preparation. Specifically, features which can strongly affect the microstructural and mechanical characterization such as hydride formation in titanium [2], amorphization of surface layers in silicon [3–6], formation of new phases and texture development in copper [7], and $\gamma \rightarrow \alpha$ phase transformation in various steels [8–11], have been reported in literature.

As Ga^+ ions can potentially alter the sample microstructure, it is important to not only understand those changes but also the conditions such as beam current, energy, effect of grain orientation etc., under which the changes occur. A thorough understanding of such microstructural alterations assumes considerable significance particularly in nuclear materials research for several reasons.

First, in the case of irradiated materials, the specific activity of the samples, which needs to be low in order to limit personnel exposure, necessitates the volume of material that can be analysed to be very small, making the use of FIB for extracting small specimens inevitable. Secondly, systematic high energy ion beam bombardment experiments have been shown to successfully capture the effects of neutron irradiation in structural materials [12]. In such a case, though FIB is an attractive instrument to prepare site-specific specimens from ion-implanted samples, the nature of the artefacts produced if any, in virgin samples prepared via FIB needs to be properly understood and characterized *a priori* so as to understand the effects due only to the ions used to simulate neutron damage, and eliminate the effects that occur due to the sample preparation using FIB.

There are two publications on the $\gamma \rightarrow \alpha$ phase transformation in different grades of stainless steels implanted with Ga^+ ions in FIB, performed primarily to understand the nature of this phase change [8,9]. Knipling et al. mainly considered the differences in the propensity of such phase transformation across alloys of varying austenitic stability [8], while Basa et al. studied the phase change in

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a duplex stainless steel [9]. Both of the studies suggest that the transformation is driven by the chemical change in the target material. Specifically, it is claimed that the $\gamma \rightarrow \alpha$ transformation occurs because of the local enrichment of gallium, which is a ferrite stabilizer.

Phase transformations have also been observed in steels of various starting crystal structures when implanted with different types of ions [13–19]. Phosphorous and antimony ion implantations have been observed to induce $\gamma \rightarrow \alpha$ transformation in stainless steel [16–18,20,21]. This transformation was also detected in neutron-irradiated and helium-implanted austenitic stainless steel [22]. It was seen that the martensite structure, which formed after deformation, transformed to ferrite phase after neutron irradiation [23]. All of these observations suggest that such transformation is primarily related to a relief mechanism of stresses developed by the accumulation of irradiation damage. A reverse transformation was also observed when the ferrite phase was bombarded with nickel or nitrogen ions [23], suggesting the role of chemical changes and related diffusion mechanisms in such a phase transformation.

Within the context of understanding the $\gamma \rightarrow \alpha$ transformation in austenitic steels occurring due to Ga^+ ions during FIB milling, previous studies have focussed on characterizing the surface of the transformed region using the electron backscatter diffraction (EBSD) technique [8,9]. Although the absence of orientation gradients, i.e. strains, in the EBSD maps shown in Ref. [9] suggests that the transformation is chemically driven, it is necessary to map the spatial distribution of gallium along the thickness of the transformed layer to demonstrate the validity of that theory. Without such analysis it is difficult to state with certainty the reason for this transformation, as earlier studies on austenitic stainless steels implanted with ions that do not stabilize ferrite, also exhibited similar transformation [13–19], which supports the strain arguments for the origin of the transformation.

The $\gamma \rightarrow \alpha$ phase transformation in 316L specimens prepared via FIB has not been reported in open literature either because of a much higher stability of γ than in other grades of austenitic steel, and hence the transformation occurs under severe milling conditions under which FIB specimens are usually not prepared, or because it has not been systematically studied, albeit observed to occur. The primary motivation of the current work is to understand the origin of the $\gamma \rightarrow \alpha$ phase transformation in this material. In addition, the effect of γ orientation on propensity of the transformation, variant selection in α and their evolution, and the effect of sputtering rate and topographical evolution have also been studied systematically.

2. Experimental methods

The material used in this study is a forged 316L austenitic stainless steel. Two specimens, A and B were considered for this study. Specimen A had an initial grain size of ~ 10 μm while specimen B had an initial grain size of 50 μm. Since we needed a larger grain size for making many FIB implantations in the same grain, specimen B was annealed for 66 h at 1100 °C under ambient atmosphere, which resulted in most grains having a size >500 μm. The samples were then ground and polished using standard metallographic preparation techniques for Ga^+ ion implantation and Electron Backscatter Diffraction (EBSD) examination.

Focussed ion beam (FIB) was used to implant Ga^+ ions with 1 nA and 7 nA currents at 30 kV on the sample surface using an FEI Quanta 3D dual beam FIB. Microstructures were characterized using different techniques. Initially, orientation imaging using EBSD technique and Scanning Electron Microscopy (SEM) were performed on the implanted regions using a FEI Quanta 650 at 20 kV operating voltage. In order to identify any transformed regions after Ga^+ implantation, maps with a step size of 1 μm were recorded while local misorientations in the transformed regions in specimen B were obtained using a step size of 100 nm.

Specimens for cross sectional transmission electron microscopy (TEM) analysis were prepared from implanted regions of the grain covering both implanted and unaffected material, with FEI Quanta 3D dual beam FIB using the standard cross section preparation method with a final cleaning step at low keV ion beam [24]. TEM images were acquired along with orientation mapping using Nanomegas ASTAR Precession Electron Diffraction - Orientation Imaging Microscopy (PED-OIM) on a FEI Tecnai F30 Transmission Electron Microscope (TEM). A step size of 2.5 nm was used for acquiring the OIM maps. Spatial distribution of gallium along the transformed layer thickness was analysed using Energy Dispersive Spectroscopy (EDS) with Scanning TEM (STEM) in probe spherical aberration corrected FEI ChemiSTEM™ Titan operated at 200 keV and 0.6 nA. STEM High Angle Annular Dark Field (HAADF) images were obtained from the same instrument. The inherent assumption in using electron beam based probing techniques (SEM, TEM) for analysing the implanted samples is that there will be very less diffusion of atoms during the electron beam exposure. This is valid since the amount of energy transferred to the material during electron beam exposure is relatively less and the amount of local heating is very less in bulk samples compared to the ion beam exposure. The depth profile of implanted regions was analysed using Keyence VK X 210 series Laser confocal microscope.

3. Gallium ion penetration depth modelling

The penetration of gallium ions in 316L steel was modelled using SRIM [25] and MDRANGE [26]. In both these codes, an interatomic potential, *V(r)*, describes the interactions between the atoms in the target albeit the models used for ion-solid interactions are different. Calculations in SRIM are performed by the binary collision approximation where in the interactions are treated as a series of two-body collisions. The scattering integral, which takes into account the impact parameter, is solved for each collision. Since the impact parameter is chosen from a probability distribution (based on the composition and atomic density of the target material) rather than from the crystal structure of the target, the calculations are performed in amorphous materials. For the ion range calculations of 30 keV Ga^+ ions in 316L austenitic steel using SRIM, 10,000 ions were bombarded normally (at 90° to the surface) into the sample with a composition consisting of 73% Fe, 17% Cr, and 10% Ni (in weight %), with a density of 7.9 gm/cc. The interactions between the atoms are described by the Ziegler-Biersack-Littmark (ZBL) interatomic potential.

In MDRANGE, molecular dynamics (MD) algorithms are used to calculate ion ranges in a variety of target structures (crystalline, amorphous, polycrystalline, and more). In MD method, the time evolution of an ensemble of atoms is calculated by numerically solving the equations of motion of all atoms in the simulation cell. The atoms are first initialized to have random displacements from their lattice positions in the specified crystal structure. The motion of the atoms is determined from both ion-ion interactions (using interatomic potential) and ion-electron interactions (electronic stopping). For the range calculations in MDRANGE, a variable time step is used to increase the speed. The code uses a translation method rather than periodic boundary conditions to keep the dimensions of the simulation cell at minimum while ensuring the recoil atom to be present in an unaffected region of the cell. For simulating the interaction of Ga^+ ions in 316LL steel, both FCC (austenite) and BCC (ferrite) crystals with lattice constants of 3.66 Å and 2.87 Å respectively, and with a composition of 73% Fe, 17% Cr,