Methodology for observation of maraging tool steel after 3D printing using FIB and STEM mode

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Methodology for observation of maraging tool steel after 3D printing using FIB and STEM mode

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Abstract. The components of the maraging tool steel produced by 3D printing are further heat-treated after printing. It includes a stress relief annealing and precipitation hardening. During this process precipitates of Ni_3Ti and Fe_2Mo or Fe_7Mo are formed, which significantly contribute to an increase in ultimate tensile strength, hardness, and toughness. To understand the precipitation processes, it is necessary to describe the particles in detail. However, they are very small in size and cannot be examined properly by a light microscope (LM) or by scanning electron microscope (SEM). A possible way to investigate and describe these precipitates is to produce a thin lamella using a focused ion beam (FIB) in the electron microscope chamber, where scanning transmission electron microscopy (STEM) is subsequently used for observation. A lamella was prepared from the DMLS (Direct Metal Laser Sintering) printed part by an ion beam for observation in STEM mode. The experiment took place at the Zeiss AURIGA scanning electron microscope, equipped with an ion gun and also with STEM capabilities for thin samples. The lamella preparation methodology was gradually optimized to achieve sufficient resolution for observation of these very fine microstructures produced by 3D printing.

Key words: FIB, STEM, maraging steel, 3D printing

1 Introduction

Due to their properties, maraging steels are suitable materials for use in many industrial applications. These are high strength steels with low carbon content, which are mainly alloyed with nickel, cobalt, molybdenum, and titanium. Maraging steels have a very fine martensitic microstructure whose mechanical properties are achieved by suitable heat treatment, in particular by precipitation hardening [1, 2]. A detailed microscopic analysis is necessary to observe the microstructure development after heat treatment and to investigate precipitates, which are developed during precipitation hardening. A light microscope will not provide sufficiently detailed information to describe such a microstructure. Using a scanning electron microscope, it is possible to describe the structure in more detail, but for a precise description of precipitates in the microstructure, it is necessary to use a transmission electron microscope or a scanning electron microscope with the possibility of transmission mode. For this purpose, it is necessary to prepare thin samples – lamellas. A scanning electron microscope equipped with two guns (dual beam microscope) was used for this experiment – dual beam microscope. It combines two independent microscope columns, a focused ion beam (FIB) and electron beam in the same machine [3, 4].

Although the preparation of thin samples by ion beam is already an understood phenomenon, it is necessary to find suitable parameters for each material, as well as for different conditions of its processing. In this case, it was a sample of maraging steel after 3D printing, which was annealed for stress relieving and subsequently it underwent the precipitation hardening treatment [5].

2 Experimental program

The experiment was performed on EOS MS1 maraging steel, whose chemical composition corresponds to US grade 18% Ni Maraging 300, the European 1.2709 and the German X3NiCoMoTi 18-9-5 grade steels (table 1). After printing, the material was annealed for stress relieving at 820°C and subsequently precipitation hardened at the temperature of 490°C for 6 hours. To prepare a thin sample for observation in STEM mode, a small piece was taken from the material using a precision metallographic saw, which was then ground and polished. It was then attached to a stub for an electron microscope using carbon tape for subsequent processing in the SEM. Different parameters of thin specimen preparation for STEM mode using ion gun were tested on the sample, which was printed using DMLS technology. This was done on a Zeiss Crossbeam 340 dual-beam scanning electron microscope equipped with a field emission gun of the Schottky type with an electron beam resolution of 1 nm. The system also featured a Focused Ion Beam (FIB) gun, detectors of secondary and back-scattered electrons (SE, BSE), an energy-dispersive X-ray spectroscopy (EDS) detector, electron backscatter diffraction (EBSD) detector, and also provided scanning transmission electron microscopy (STEM) capabilities for thin samples which features gas injection system (GIS) equipped with platinum.

Table 1. The chemical composition of the experimental material, EOS Maraging Steel MS1 [1].

wt. [%]	C	Si	Mn	P	S	Cr	Mo	Ni	Co	Ti	Cu	Al	Fe
MS1	≤ 0.03	≤ 0.1	≤ 0.1	≤ 0.01	≤ 0.01	≤ 0.5	4.5 - 5.2	17.0 – 19.0	8.5 – 9.5	0.6 – 0.8	≤ 0.5	0.05 – 0.15	bal.

2.1 Lamella preparation

The thin sample – lamella was prepared in several steps using FIB, GIS and nanomanipulator. The liquid metal ion source of gallium (Ga^+) is used as an ion source for FIB. Gallium ions are extracted from a liquid metal ion source. The ions are accelerated by the acceleration voltage up to 30 keV. The ion emission is regulated by the extractor and the suppressor. The GIS system, in combination with FIB, allows ion-beam induced deposition of platinum on the specimen surface. After selecting a suitable location for the observation that represented all investigated microstructural constituents, the sample was tilted at 54° to begin to remove material with an ion beam. First, a platinum layer was applied to the region of interest using GIS, which serves to protect the surface layer of the sample from deterioration by both ion and electron beams. Subsequently, two trapezoids were roughly excised using a probe of 30 kV to 30 nA (figure 1a). To achieve sufficient depth, a dose factor of 15 with a dose of 400 mC / cm^2 was chosen, which resulted in approximate depth of 14 μm, which was sufficient for subsequent handling of the lamella and its fine polishing and observation. The lamella dimensions required for observation of sufficient amount of microstructural constituents was about 3x4 μm. The scan mode during coarse cutting was bidirectional with cycle mode cross-section to ensure uniform cutting of the material over the entire surface of the prepared lamella.

The next step was to reduce the lamella from the original 2.5 μm to 500 nm in several steps using a lower probe, namely 30 kV: 15 nA and then 30 kV: 200 pA (figure 1b). During this process, the lamella was rotated by ± 0.5° to obtain a perpendicular cut. In case the rotation was not performed, the lamella would expand towards the base material and would not have good material removal at its base. Subsequently, the lamella had to be separated from the base material. Then a special stub with a Cu holder could be inserted into the chamber, to which the lamella was then attached with

Ga. Lamella was first undercut into a U-shape, which served as a preparatory work to move it. The nanomanipulator was used to remove the lamella and was also attached to it using GIS and Ga. Consequently, the lamella on the nanomanipulator could be transferred to the chamber space. After inserting the stub with Cu-holder, it was moved from the nanomanipulator to Cu-holder. There the lamella was reattached with Ga and the nanomanipulator was cut off by an ion beam (figure 1 d, e, f). The release of the nanomanipulator from the lamella is useful for re-sharpening the nanomanipulator with an ion beam (figure 2).

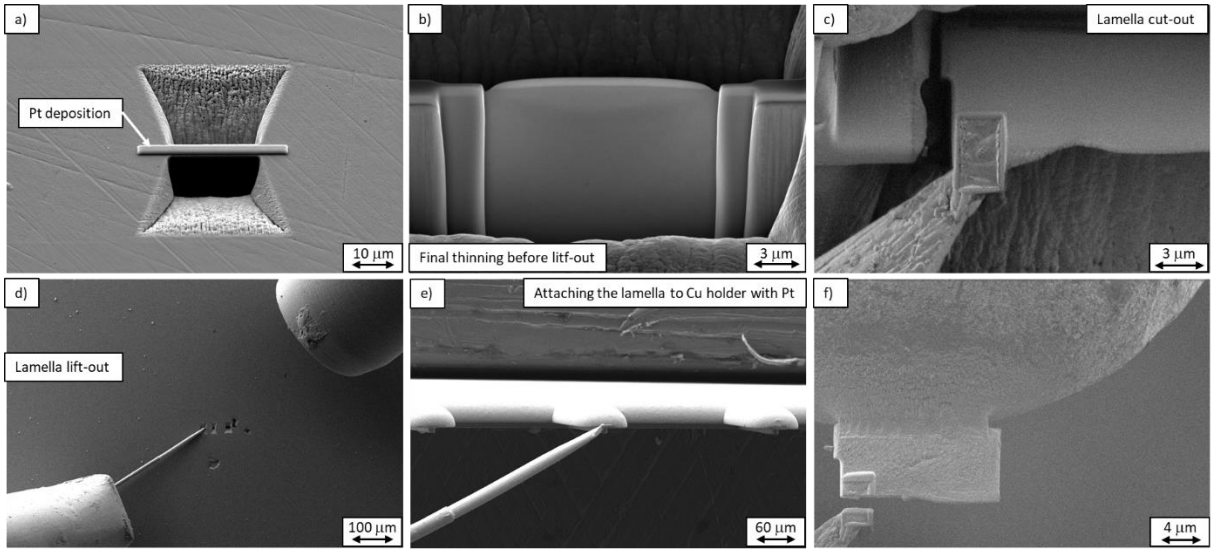

Figure 1. The scheme of lamella preparation: a) Pt deposition for surface layer protection and coarse trapezoides in the desired position on the sample, b) thinning before lift-out, c) Pt deposition to attach sample to the nanomanipulator and cut-out the lamella, d) litf-out, e) attaching lamella to the Cu holder by Pt using GIS, f) cut of the nanomanipulator.

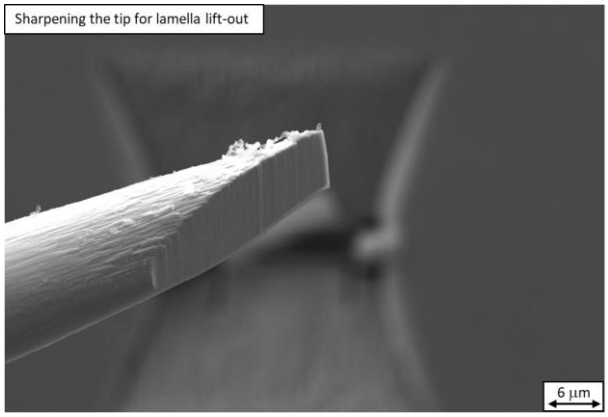

Figure 2. Sharpening of the nanomanipulator after lamella lift-out.

After shifting the lamella to the Cu-holder, further thinning of the individual layers of the material by ion beam is necessary. Lower operating parameters are used: 5 kV: 200 pA. The lamella is uniformly thinned from the previous 500 nm until a thickness of less than 100 nm is reached. The need for further thinning depends on the sample material and the acceleration voltage used for observation. For EOS MS1 maraging steel, the final thickness for observation in STEM mode was about 75 nm (figure 3).

The final step before it is possible to analyse the lamella in STEM mode is to transfer the Cu-holder with thin lamella to the special STEM holder, where the Cu-holder is placed in horizontal position in order to get its surface between electron gun and the detector of transmitted electrons.

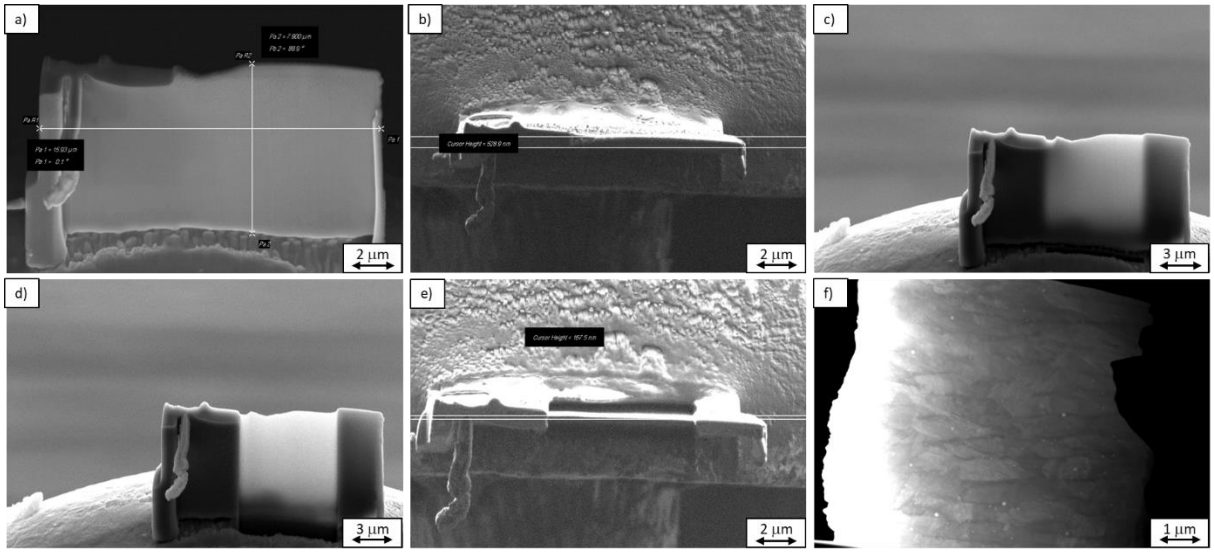

Figure 3. Continuous steps for obtaining the final dimensions of the lamella attached to the Cu-holder.

2.2 STEM observation of the lamella

The Zeiss electron microscope was equipped with 4-Channel aSTEM detector which detecting transmitted electrons in an FESEM with the annular STEM detector. The aSTEM detector collects electrons transmitted through a thin sample and it can separate unscattered and scattered electrons depending on the scattering angle. The detector constitutes of special annular arrangement of five diode segments (figure 4). The bright field (BF) segment placed in the centre of the detector detects the unscattered electrons. Around BF segment are diagonally placed two dark field (DF) segments and two segments for oriented dark field (ODF) imagining, one annular dark field (ADF) ring and the high angle annular dark field (HAADF) ring on the outer diameter to detect electrons scattered under higher angles (figure 4).

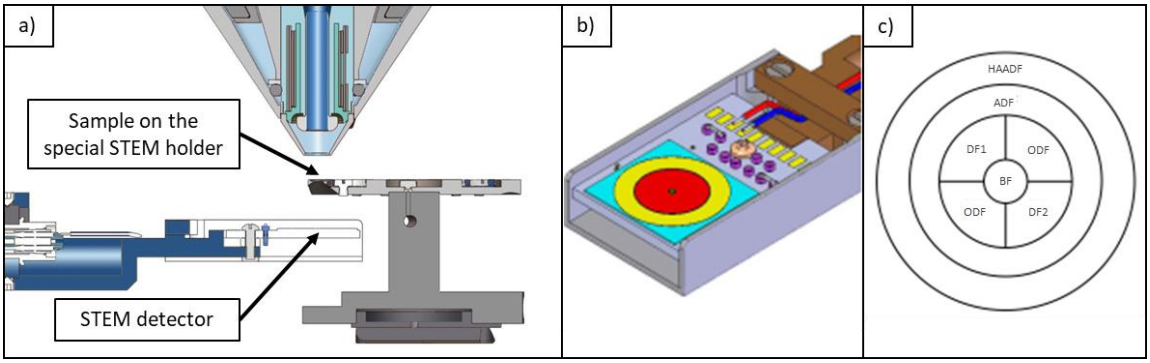

Figure 4. Arrangement for samples observation with STEM detector: (a) the sample is placed on a copper holder between the electron gun and the STEM detector, (b) head of the annular STEM detector, (c) Scheme of the semiconductor-based detector with its BF area in the centre, the four DF diodes, the ADF ring, and the HAADF outer ring [7].

With a given segments layout of the aSTEM detector, it is possible to combine signals from different segments to achieve the desired resolution and highlight precipitation or other areas of interest. The possibility of combining individual detector segments is the main advantage of a scanning electron microscope equipped with a STEM detector. By collecting and displaying multiple signals, it is possible to obtain as much information as possible from each scan. Each detector is used to highlight different points of interest in the structure of the material (figure 5). The BF detector includes a transmitted beam and is, therefore, suitable for highlighting holes that appear bright in this display. In contrast, the DF detector eliminates the transmitted beam, and the holes in this regard appear dark. An ADF detector that has the form of a ring forms an image by collecting scattered electrons similar to a DF detector in TEM. But ADF in STEM does not use an aperture to separate the scattered electrons from the main beam. The HAADF detector is, as the ADF detector, a ring, but the hole in its center is significantly larger than that of the ADF detector. Thanks to this, it detects electrons that are incoherently scattered to large angles. The result is an image called Z-contrast [8, 9]. It is obvious from the microstructure images that the basic matrix of the experimental material is formed by martensitic needles, among which spherical-shaped precipitates occur. Due to the shape and size of the precipitates and heat treatment of the material, most of these precipitates are Ni_3Ti or $Ni_3(Ti, Mo)$. The larger diameter particle that is visible in the lower-left corner of the HAADF image (figure 5d) is probably a Fe_7Mo6 precipitate that typically reaches a size between 10 - 30 μm. Precise determination of observed precipitates is possible only by using TEM and diffraction pattern, or by measuring chemical composition and stochiometry. The bright areas in figure 5e are retained austenite [10].

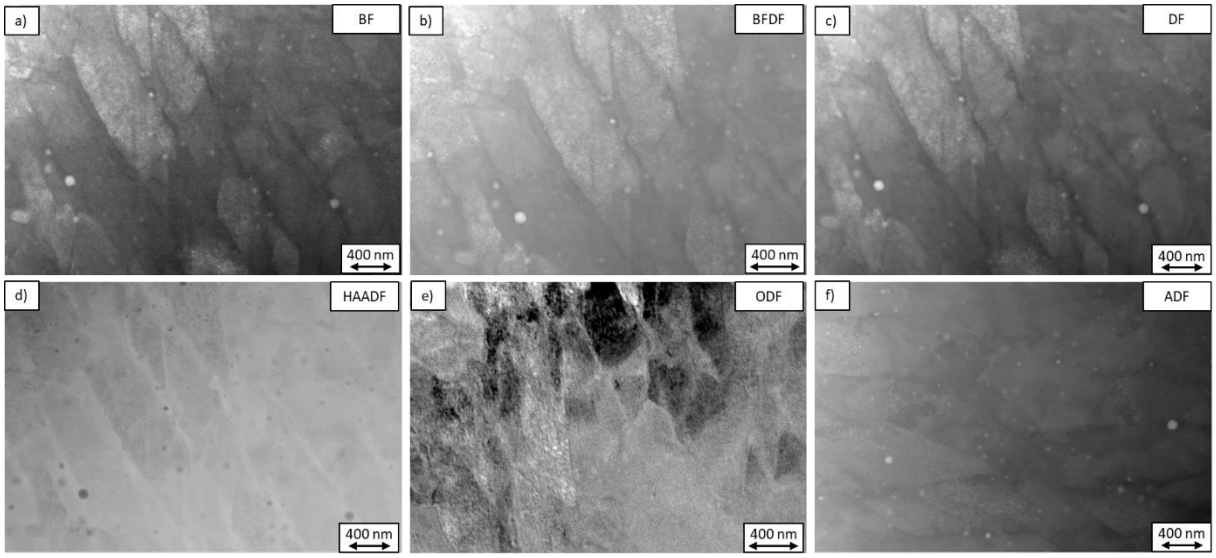

Figure 5. Different modes for sample observation.

3 Conclusion

Using an ion beam scanning electron microscope, a gas injection system and a detector for field emission scanning aSTEM, it was possible to prepare and observe a thin lamella from a maraging steel sample printed by the DMLS method. The lamella was prepared in several steps using various probes of the ion beam. Since the structure of the material is unconventional and very fine after 3D printing, it was necessary to continuously optimize the parameters to obtain sufficiently high-quality images in the transmission mode, which makes it possible to describe all the microstructural elements adequately. It has been found that to achieve lamella with dimensions of approximately 4 x 3 μm it is suitable to use probe 30 kV: 30 nA for coarse milling up to a thickness of 2.5 μm, followed by 30 kV: 15 nA and 30 kV: 200 pA for finer milling to a thickness of approximately 500 nm. After the lamella is moved to the Cu-holder, a 5 kV: 200 pA probe was applied for fine polishing to less than 100 nm thickness. In this state, the specimen on the Cu-holder were transferred to a special STEM sample holder, which was located between the electron gun and the aSTEM detector in the microscope chamber, allowing the transmission mode to be applied.

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