Evaluation of residual stresses by use of dual beam SEM

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Three-Dimensional Electron Backscatter Diffraction (3D-EBSD) technique is based on incorporating a focused ion beam (FIB) column into a conventional scanning electron microscope (c-SEM). Such an instrument is thus referred to as FIB-SEM. The measurement is carried out by repetitive iteration of two basic steps: cross-section milling using the ion beam and acquisition of electron backscatter diffraction data (which is a starting point to crystal orientation determination) using the electron beam. The 3D orientation microscopy enables comprehensive microstructural characterization, including e.g. grain morphology, grain boundary geometry and pores distribution.

Combined dual beam SEM facility is also an exciting tool for residual stress evaluation in the microscale. In the recent years, there is a growing interest in FIB-SEM investigations of stress distribution in different range of materials, and this field of electron microscopy is developing rapidly nowadays. Some researches push forward the experimental technique when the others focus on post-processing data interpretation. We concentrated on delivering the results of micro-scale stress evaluation by use of FIB-SEM and further comparison with XRD measurements. The basic material used in the present study is 30 μm thick, 25 mm wide ribbon prepared by single wheel melt spinning. The $Fe_{80}Si_{11}B_9$ alloy, amorphous in the as-received condition, was supplied by Allied Signal Corp. of Morristown, New Jersey. The pulsed laser interference heating (PLIH) was carried out using a Q-switched Nd:YAG with laser wavelength of 1064 nm, frequency of 10 Hz, pulse time of 10 ns and 90 or 120 mJ laser energy and a variable number (from 50-500) of consecutive laser pulses.

It should be pointed out that the laser beam has a Gaussian distribution of intensity which results in inhomogeneous heating across the laser beam diameter. The laser energy density decreases exponentially in the radial direction and has a maximum value at the centre of irradiated dots. As there is the highest temperature, it is indeed much more deeply melted than the other regions (Fig. 1a). The large temperature gradients achieved with localized laser heating can lead to rapid self-quenching of the material, trapping highly non-equilibrium structures. Also, the rapid generation of large temperature gradients can induce thermal stresses and thermo-elastic excitation of acoustic waves [1-3]. Un-melted regions experienced a gradual decrease in temperature, which finally resulted in partial crystallization of the amorphous substrate. Non-processed material between the irradiated spots remained in the amorphous state.

We share our experience in the field of residual stress measurements by application of the FIB-SEM technique from the conceptual stage; first experiments and data analysis to the variety of approaches, tips and hints, up to correct results confirmed by the XRD technique. Custom made program was elaborated for digital image correlation (DIC) based on *Wolfram Mathematica* package. Correctness of results was checked also based on the literature reports.

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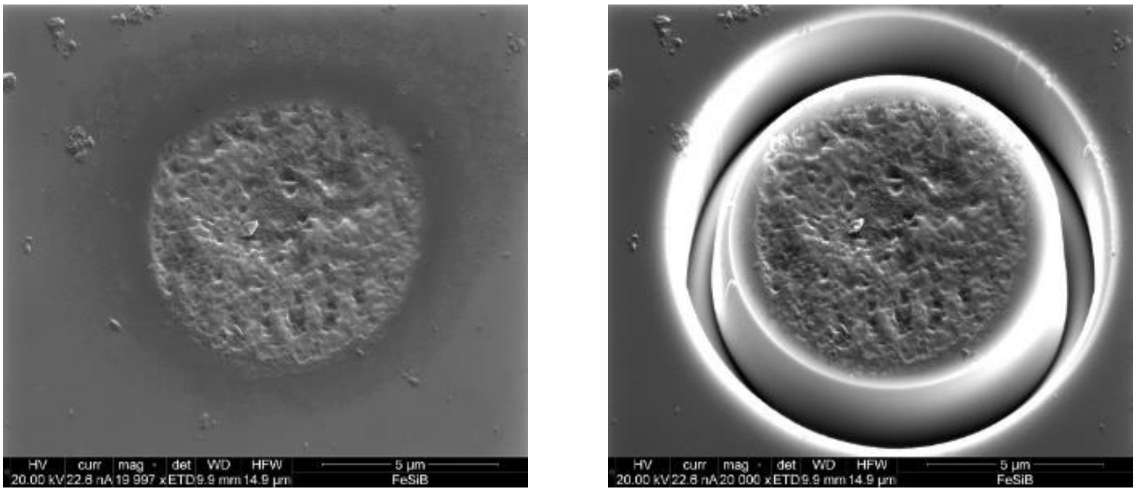

FIGURE 1. SEM images obtained during FIB SEM residual stresses measurement in the subsurface volumes of the $Fe_{80}Si_{11}B_9$ sample. Different approaches were used to evaluate the strain relaxation.