Comparison of three-dimensional atom probe measurements of an Fe-Co-Mo-Cr alloy using laser and voltage pulsing

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The three-dimensional-atom-probe (3DAP) provides 3D microstructural information on a nearly atomic scale. Because of this high resolution atom probe is a good method to investigate microstructural phenomena like spinodal decomposition [1], early stages of precipitation [2], nanocrystalline materials [3], and segregations at interfaces [4] etc. The method is based on the field evaporation generated by a combination of a high DC standing voltage and nanosecond long high voltage pulses applied to a tip like shaped specimen with an end radius of about 50-100nm [3, 5, 6]. For propagation of high voltage pulses good conductivity of the specimen is required, limiting the application of this technique to metallic materials and highly doped semiconductors [6, 7]. An other possibility to produce field evaporation is to use a combination of a high D.C. voltage and laser pulses [8]. In the laser assisted 3DAP the ion emission is triggered by laser pulses and the ions are accelerated by the standing field applied to the specimen [9, 10].

The laser assisted field evaporation brings several advantages compared to voltage pulsing. In addition to providing the possibility to analyse poorly conductive materials, it allows a larger field of view of the instrument. Performing investigations at lower standing voltage fields and therefore lowering the mechanical stress on the specimen very brittle materials can be analyzed with a good rate of success [10].Laser assisted atom probe is an emerging characterization method, hence, the influence of the laser pulse on the analyzed material is not entirely understood [6]. The aim of this work is to highlight effects which have to be taken into consideration using laser assisted field emission. Experiments were conducted on an Fe-Co-Mo alloy. These alloys show, like precipitation hardend aluminium alloys, a strong increase (about 30HRC) in hardness during ageing after a solution heat treatment. This increase in hardness is caused by the precipitation of nanometer sized intermetallic precipitates [11]. 3DAP is an invaluable tool to characterize size, shape, and chemical composition of these nmsized precipitates. To investigate the influence of the different ways to reach field evaporation (voltage and laser pulsing) on the measurement results experiments were conducted in both modes.

Atom probe samples were mechanically cut from the heat treated samples (12 x 0.3 x 0.3 mm^3) and electropolished using standard 2-step techniques [7]. Atom probe experiments were carried out on an IMAGO-LEAP 3000X-HR System at the University of Leoben, Austria. Atom probe data acquisition was performed under ultra high vacuum conditions at a specimen temperature of 50 K with a pulse repetition rate of 200 kHz and a pulse fraction of 20 % for the voltage mode. In laser mode a pulse repetition rate of 200 kHz and a laser energy of 0.2 nJ was applied.

Figure 1 shows 3D elemental maps of a specimen measured in laser mode. As can be seen density fluctuations of Mo and Cr, indicating precipitates are obvious. A comparison of obtained mass spectra given in Fig 2 shows considerable differences between voltage and laser mode. The mass spectra achieved from measurements in laser mode exhibit remarkably broader peaks than the mass spectra obtained from voltage mode measurements. Due to this peak broadening peak overlapping leading to variations in the chemical compositions can

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occur. The performed comparison of laser mode to voltage mode indicates strong influence of laser energy and sample temperature on the exhibited spectra and the spatial resolution.

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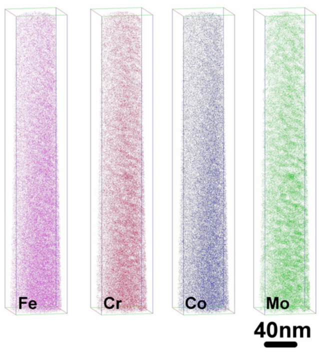

Figure 1. 3D LEAP reconstruction of a Fe-Co-Mo-Cr alloy using laser assisted field evaporation.

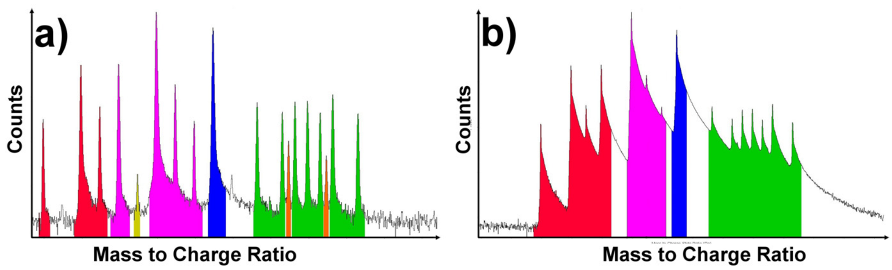

Figure 2. Mass spectra of iron- (pink), chromium- (red) cobalt- (blue) and Molybdenum- (green) ions measured in a) voltage mode and b)laser mode.

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