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Focused ion beam sample preparation of mechanically alloyed iron powders for atom probe tomography

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Higher temperature strength in steels can be achieved with addition of finely distributed Yttrium oxides by mechanical alloying. Nevertheless, the strengthening mechanism during mechanical alloying is still not clearly understood. In order to clarify the effects of Y and O distribution during the alloying process, 4 different manufactured powders were analysed.

Two different iron-rich powders have been analysed, see figure 1. The powder was mechanically alloyed for 12h and 48h using a Certony™ attritor. The iron powder was produced by water atomization and was mixed with 0.5 wt% yttria powder with a d_{90} of 1.16 µm. The chemical composition is presented in table 1.

For the characterization of the different powder conditions, atom probe tomography (APT) has been used [1]. For APT measurements needle-shaped specimens are needed, with radii between 30 to 100 nm on the tip and an angle near to or lower than 10°. Traditional methods as chemically or electrochemically polishing [2] are impossible to use in powder material. Therefore, the method for preparation of needle-shaped tips for powder samples via focused ion beam (FIB) was chosen [3, 4]. A single grain of each powder was selected for a thick lift out in a wedge form and a platinum layer was deposited on the interesting zone, first via electron deposition and afterwards with ion deposition, to minimize gallium implantation. Multiple specimens were cut from the thick lift out wedge and welded on a micotip coupon, which provides the pre-tips for the APT measurements. Afterwards, the following annular milling at 30 kV, 5 kV and 2 kV were the last process, for the preparation of the APT tips free of Gallium implantation. The atom probe investigations were completed in a LEAP™ 3000X HR of Cameca™ with a Local Electrode system. The analyses were performed in voltage mode at 60 K in ultra-high vacuum. A pulse fraction of 15 % and 20 % was used. The atom probe measurements were evaluated with IVAS 3.4.3 software. The analysed volume was about 25x25x150 nm^3 and the different material conditions were compared.

The atom probe investigations reveal a clustered arrangement of Y and O atoms during mechanical alloying; see figure 2 a) and b). Yttrium atoms together with oxygen, rearrange during mechanical alloying to form clusters. Aluminium and manganese enrichments (see figure 2 c) and d)) could be also detected, which suggests that diffusion processes are active during the mechanical alloying process. These clusters are not only remnants of the Y_2O_3 powder, yttrium and oxygen dissolve in the matrix to rearrange afterwards at favourable sites as lattice defects, vacancies or dislocations to form clusters.

By using focused ion beam, needles-shape specimens for atom probe tomography could be prepared for two different grain size powder samples after 12 and 48 h of mechanical alloying. The atom probe investigations reveal fine distributed yttrium oxide clusters as well as yttrium dissolved in the matrix. Moreover enrichments of alloying elements as aluminium and manganese reveal that diffusion processes are active during mechanical alloying clarifying the mechanism during this process.

1. W.R. McKenzie, E.A. Marquis and P.R. Munroe, “Microscopy: Science, Technology, Applications and Education”, ed. A. Méndez-Vilas and J. Díaz (2010) p. 1800.
2. M.K. Miller, A. Cerezo, M. G, Hetherington and G.G.W. Smith, “Atom Probe Field Ion Microscopy”, ed. R.J. Brook, A. Heuer, T.J. Marks, D.A. Smith, A. Cheetham, S.P. Hirsch, J. Silcox, M.V. Tirrell, V.Vitek (1996) p.476.
3. M.K. Miller, K.F. Russel, K. Thompson, R. Alvis and D. J. Larson, Microscopy and Microanalysis 13 (2007), p. 428.
3. M.K. Miller and K.F. Russel, Ultramicroscopy 107 (2007), p. 761.

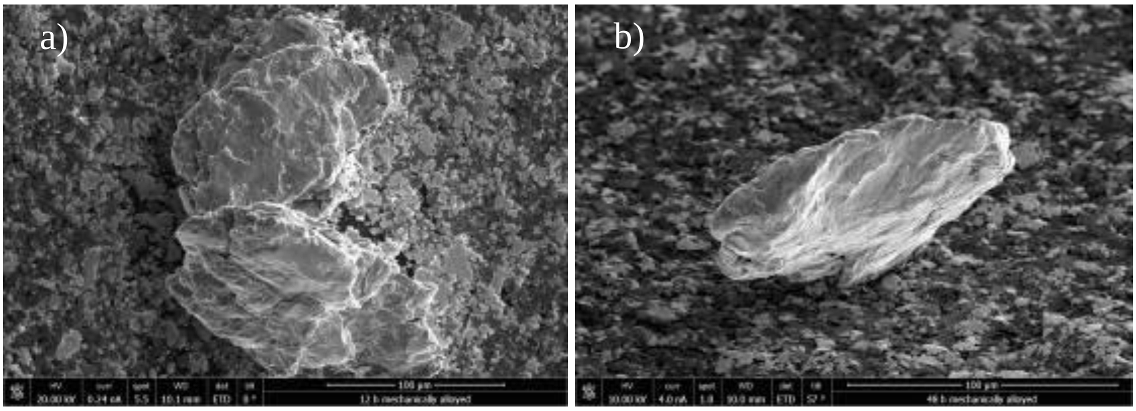

Figure 1. Powder after 12 h mechanically alloyed (a) and after 48 h mechanically alloyed (b).

	Fe	Al	Mn	Cr	Ni	Mo	W	Y_2O_3
12 h mechanically alloyed	bal.	0.34	0.14	0.09	0.05	0.01	0.8	0.12
48 h mechanically alloyed	bal.	0.1	0.16	0.24	0.1	0.01	0.05	0.13

Table 1. Chemical composition of the of the analyzed powders (at.%).

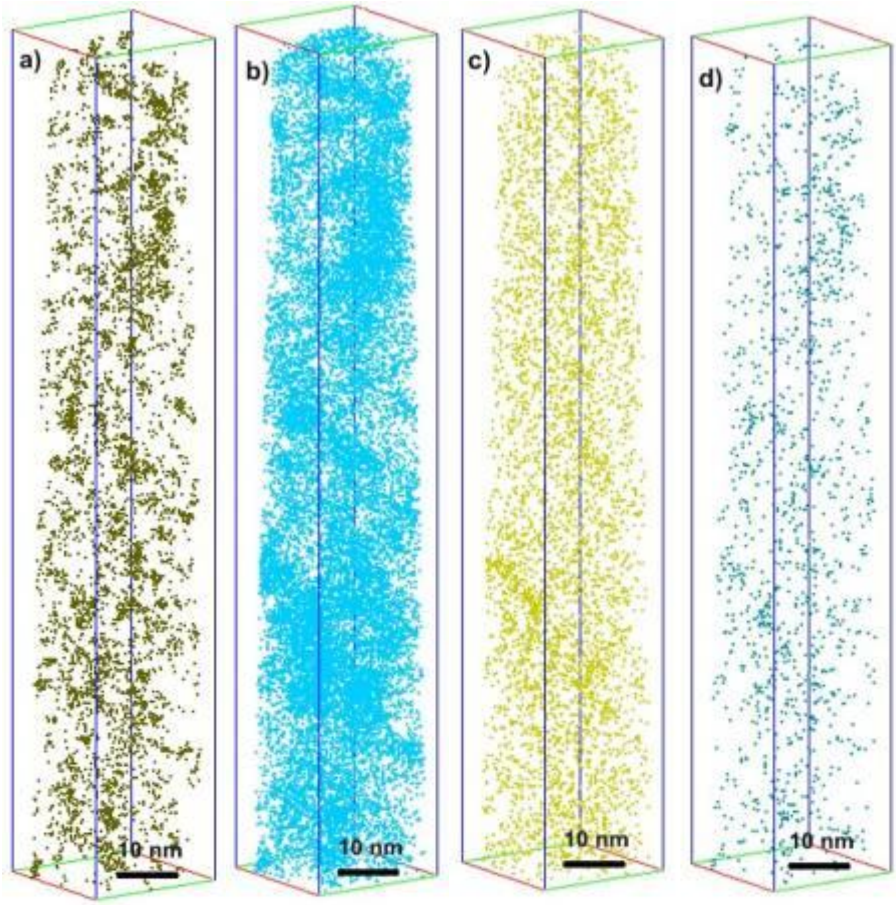

Figure 2. 3D atom maps of the 48 h mechanically alloyed condition. a) Y; b) O. 3D atom maps of the 48 h mechanically alloyed state, showing enrichments of Al and Mn at the Y-O clusters c) Mn and d) Al.