Functional Materials

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SEM study of microstructure of Fe-Mo powders prepared by ball milling

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Mechanical alloying (MA) is a way of preparing various kind of materials in the form of powders. It is a high-energy processing (e.g. ball milling) where mechanical energy of high speed rotation is transferred to the material. During this process the induced strains lead to repeated fracturing and cold welding of source materials. As a result the alloy powder is formed from blended elemental powders [1]. The process of mechanical alloying with involvement of high energy transfer is able to produce various unstable forms of materials, such as amorphous, metastable and oversaturated alloys. Despite an extensive experimental research [2,3] there is still a lack of understanding of complex physical and chemical mechanisms of the structure and phase formation during MA. This work is focused on the study of microstructure of Fe-Mo powders as depending on the milling conditions and further thermal treatment.

A Fritsch Pulverisette 7 premium line planetary ball mill was used to prepare the samples. The starting materials for ball milling were crystalline Fe (150μm) and Mo (5μm) powders of high purity (99.8%). They were at first annealed at 873K for 100 hours and slowly cooled down in the mixture of Ar and H_2.Then the powders in the ratio $Fe_{80}Mo_{20}$ were placed in two vials and mixed without balls at 150rpm for 1 minute. After that, 10pcs of balls of 10mm in diameter were added in each vial. The material of vials and balls was zirconium oxide. One of the closing lids was equipped with sensors for temperature and pressure monitoring during the milling in air. The other one enabled milling under nitrogen gas. The speed of rotation was 450rpm, the ball-to-power ratio was 10:1 and the maximum milling time was 250 h. To avoid undesired sample overheating every 60 min of milling were followed by a 60min pause. Powder samples were taken out at preselected times of milling for microstructural analysis. Samples after the final milling time of 250 h were optionally annealed at vacuum furnace at temperatures ranging from 673 to 823 K.

An X'PERT-PRO diffractometer with Co Kα radiation was used to study the occurring structural changes during milling. A TESCAN LYRA 3XMU FEG/SEM×FIB scanning electron microscope (SEM) working at accelerating voltage of 15 kV with an Oxford Instruments energy dispersive X-ray (EDX) analyzer X-Max 80 and a Philips CM12 STEM transmission electron microscope (TEM) with an EDAX EDX and Phoenix software were used to follow the surface morphology and microstructure.

X-ray diffraction analysis yielded the volume fraction of coexisting phases and respective mean diameters of coherently scattering crystal regions. An essential decrease of crystal regions size occurs during initial 80 h of milling. It turned out that Mo begins to dissolve in Fe and vice versa already after 10 h of milling. The samples annealed for 250 h both in air (A) and nitrogen (N) consist of dominant (nearly 90 %) bcc-Fe(Mo) phase and the residual bcc-Mo(Fe) phase. The A and N samples differ in the Mo content dissolved in Fe. According to lattice parameter measurements, in the A sample 14.4 at.% Mo was detected in bcc-Fe(Mo), while in the N sample approximately 11.4 at.% Mo was found. The crystallite size was about 6 nm for bcc-Fe(Mo) and 8 nm for bcc-Mo(Fe) phase in both samples.

TEM observations (Figure 1) were made on powder samples deposited on holey carbon films. They show compact agglomerates with internal structure consisting of small crystal regions, the size of which correlates well with the results of X-ray diffraction. Point EDX analyses in TEM reveal compositional variations inside the agglomerates [4]. Both X-ray and EDX measurements confirm that the Mo content in the bcc-Fe(Mo) and the Fe content in the bcc-Mo(Fe) solid solutions exceed substantially the equilibrium solubility limit in both A and N samples.

SEM observations document the powder size decrease in the course of milling (Figure 2). It should be noted that in SEM micrographs we observe agglomerates and only TEM can visualize their internal microstructure. Figure 3 displays the smallest agglomerate size after milling for 250 hours. Annealing at relatively low temperatures does not substantially affect the overall look of studied powder samples from the viewpoint of agglomerate and crystallite size. Extensive EDX analyses in SEM after various heat treatments found a reasonable amount of Zr in all measurements, originated from the vials and

balls. Small ZrO_2 peaks were detected also in X-ray diffractograms. Point EDX analyses from single agglomerates (though encompassing a large number of crystallites each) have a large scatter. Average values are 76Fe-16Mo-8Zr (at. %) for A samples and 76Fe-17.5Mo-6.5Zr for N samples [5].

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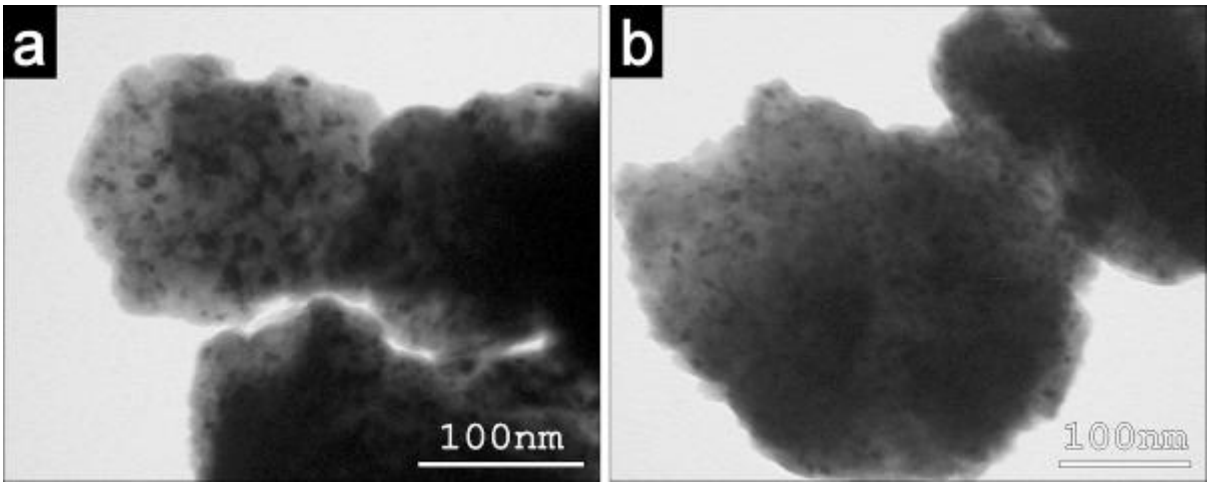

Figure 1. TEM micrographs of powder particles after 250 hours of milling in air (a) and in nitrogen (b).

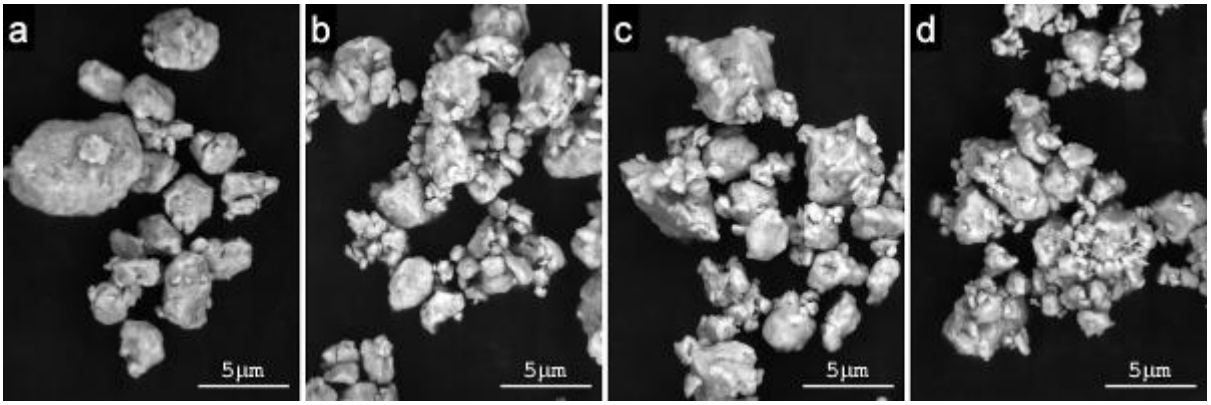

Figure 2. SEM micrographs of powders (backscatter electron images) in selected intermediate stages of milling: 60 hours in air (a), 60 hours in nitrogen gas (b), 120 hours in air (c) and 120 hours in nitrogen gas (d).

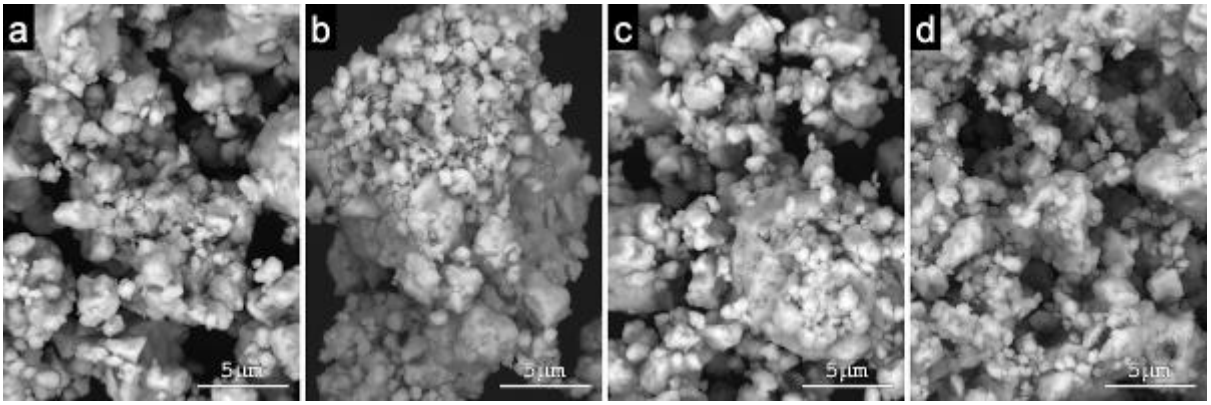

Figure 3. SEM micrographs of powders (backscatter electron images) after 250 hours of milling (in either air or nitrogen) followed by annealing at selected temperature: air, 723 K (a), nitrogen, 723 K (b), air, 773 K (c), nitrogen, 773 K (d)