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PHASE TRANSFORMATIONS IN M2 STEEL POWDERS SUBJECTED TO BALL MILLING

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Abstract. The mechanical alloying process of M2 high speed steel resulted in the formation of the powders with average particle size about 4 μm and round-in-shape morphology. The X-ray diffraction studies revealed the formation of nanoferrite characterized by the values of crystallite size and lattice strain of 12 nm and 1%, respectively. The Mössbauer measurements allowed to distinguish between ferrite and alloyed ferrite and to estimate the content of both phases after 100 h of mechanical alloying as 20 and 71%, respectively. Moreover, the content of iron containing carbides was found to be 9%, most probably of the M_7C_3 and/or $M_{23}C_6$ types. Differential scanning calorimetry measurements revealed two exothermic effects during the heating of the mechanically alloyed powders, corresponding to ferrite lattice strain decrease and carbide and ferrite crystallite growth, respectively.

1. INTRODUCTION

As the most widely applied materials, steels have been well studied for many years. Many processing methods have been applied to improve their structure and properties. It is expected that further improvement of steels can be achieved by fabrication of nanocrystalline structure. Various methods are used to produce alloys with nanoscale crystallites, like equal channel angular pressing or high pressure torsion. Among these methods ball milling is regarded as an effective way for obtaining nanocrystalline materials. However, decreasing of crystallite size during ball milling of steel powders is usually accompanied by phase transformations of the material studied. The phase transformations in steels subjected to ball milling have been studied recently. Nanoferrite formation and strain-induced-ferrite transformation in austenitic stainless steel were reported [1,2]. Aging the nanocrystalline ferrite resulted in re-precipitation of fine cementite particles retarding the grain growth of nanoferrite, leading to the formation of composite of nanocrystalline ferrite and cementite. The nanocrystalline ferrite revealed a totally different morphology, microstructure and mechanical properties from the work-hardened ferrite. The formation of martensite in ball milled 316L austenitic steel was also observed as a result of ball milling [3]. The ball milling process of carbon tool steel powders led to the dissolution of cementite and formation of nanoferrite, characterized by crystallite size and lattice strain of 10 nm and 1%, respectively [4]. The number of papers on structural transformations in ball milled high speed tool steel powders is very limited. The formation of nanoferrite (‘ordinary’ ferrite and alloyed ferrite) and dissolution of carbides was reported [5]. There are no reports on an attempt of obtaining nanocrystalline high speed tool steel via mechanical alloying (MA), i.e. from the powder

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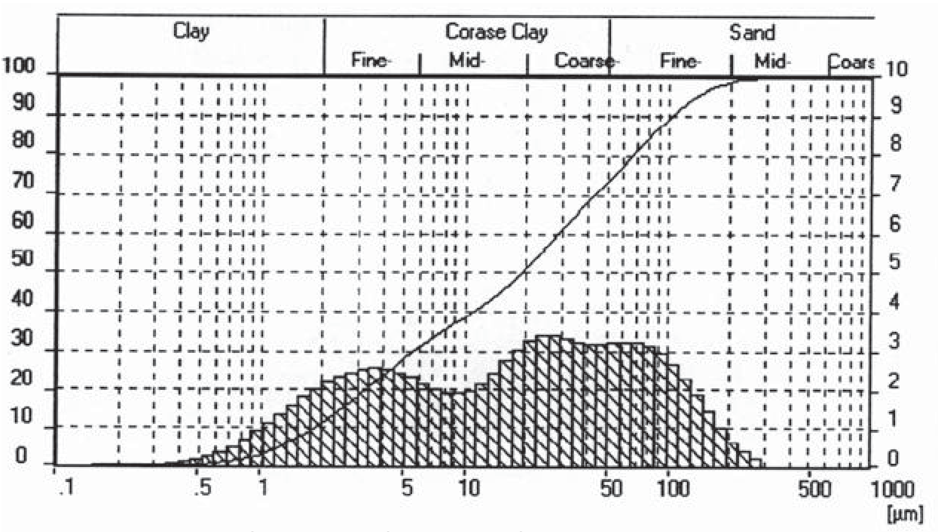

Fig. 1. Particle size distribution of M2 steel after 100 h of mechanical alloying.

mixture of pure elements. Therefore, the aim of this work was to study the phase and structural transformations in high speed tool steel powders produced by mechanical alloying technique.

2. EXPERIMENTAL

The powders of pure elements (ABCR, Germany and Pure Chemicals, Japan) with a purity better than 99.7% and particle size below 75 μm, were mixed in the proportion corresponding to the chemical composition of M2 high speed steel, i.e. 0.85% C, 5.48% Mo, 6.20% W, 0.28% Mn, 4.24% Cr, 0.29% Si, 1.98% V, and Fe as a balance. After mixing the powders were subjected to mechanical alloying for maximum 100 h. Fritsch P5 planetary ball mill equipped with hardened steel vials and balls was used. The mill operated at 250 rpm. The mass of powder was 10 g and ball-to-powder weight ratio was 10:1. All powder handling was performed in a glove bag under an argon atmosphere. Laser analyser Fritsch Analysette 22 was employed for particle size distribution determination. Particle size and morphology were also studied by scanning electron microscopy (SEM). Hitachi S 3500 N unit was used for this purpose. X-ray diffraction (XRD, Philips PW 1830, CuKα radiation) was applied for phase analysis, determination of lattice parameters of phases observed as well as for crystallite size and lattice strain calculations using Hall-Williamson method [6,7]. Differential scanning calorimetry (Perkin Elmer DSC 7) allowed thermal effects registration during heating the powders. The samples were continuously heated within the temperature range from 50 to 720 °C applying the heating rate of 20 °C·min^{-1}. Transmission Mössbauer spectra were measured at room temperature using a 25 mCi ^{57}Co-in-Rh source. The relative contents of the phases present in the samples were calculated as a ratio of the area of the relevant subspectrum to the total spectral area, assuming similar Debye-Waller factors for each phase. Isomer shifts were given with respect to the standard α-Fe foil.

3. RESULTS AND DISCUSSION

Fig. 1 shows the size distribution of the powders after 100 h of MA. A bimodal distribution centred around 4 and 20 μm is observed. However, SEM examinations reveal (Fig. 2) that 4 μm or even less is a true single particle size, while 20 μm is a size of the agglomerates formed during mechanical alloying. SEM investigations also show that independently of the shape of the starting powders, the alloyed powders after 100 h of the processing are round in shape.

Fig. 3 shows a sequence of XRD patterns registered for the samples after increasing milling times. The intensity of diffraction lines of alloying elements gradually decreases testifying the formation of ferrite. However, even after 100 h of MA residual lines of not completely dissolved Mo and W are visible. Simultaneously, the observed significant broadening of ferrite lines may testify the formation of nanostructure. The calculated values of crystallite size and lattice strain of nanoferrite are presented

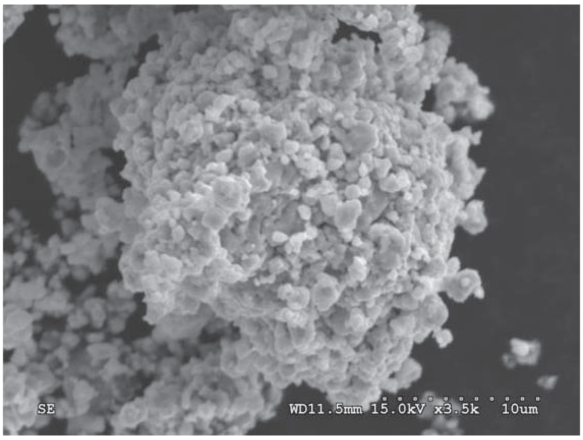

Fig. 2. SEM morphology of M2 steel powders after 100 h of mechanical alloying.

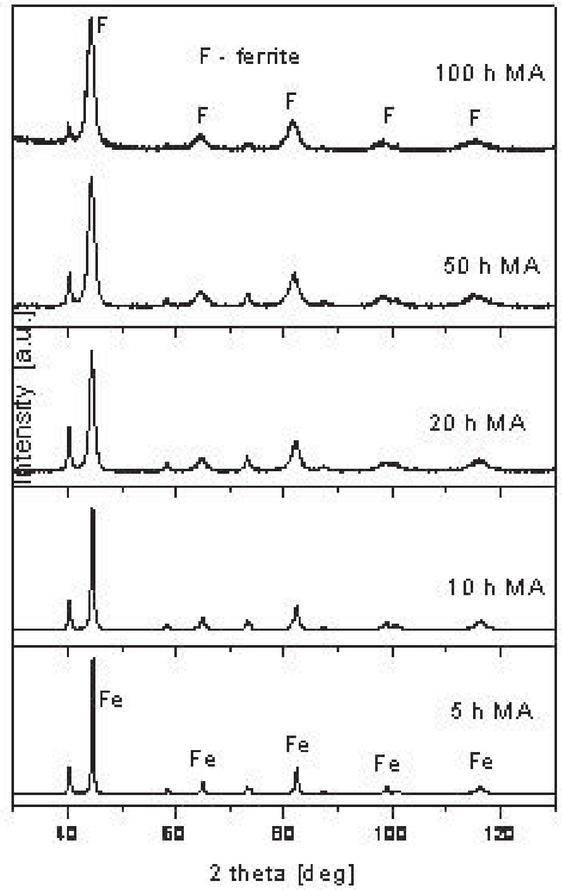

Fig. 3. A sequence of XRD patterns of the samples after increasing milling times.

in Fig. 4 and 5, respectively. After 100 h of MA the mean crystallite size goes down to about 12 nm and the lattice strain reaches 1%. Both values are typical for mechanically alloyed metallic materials. The alloying process and formation of ferrite is accompanied by the increase of the lattice parameter from the value of pure Fe (0.2866 nm) up to 0.2882 nm, as shows Fig. 6.

The Mössbauer spectra of selected samples prepared by mechanical alloying of pure elemental powders are collected in Fig. 7. There are three components fitted in the spectrum of the sample milled for 100 h: (i) a sextet with hyperfine field H_{hf} of 33.6 T originating from the ferrite phase, (ii) a sextet with broadened lines and reduced hyperfine field (H_{hf} = 29.6 T) related to the alloyed ferrite and (iii) a quadrupole doublet with quadrupole splitting of 0.55 mm/s and isomer shift of 0.08 mm/s. The last component, paramagnetic one, can be attributed to iron containing carbides, most probably of the M_7C_3 and/or $M_{23}C_6$ types. A possible distinction between different carbides types is offered by the isomer shifts only ($\delta_{M6C} < \delta_{M23C6} < \delta_{M7C3}$). However, these differ-

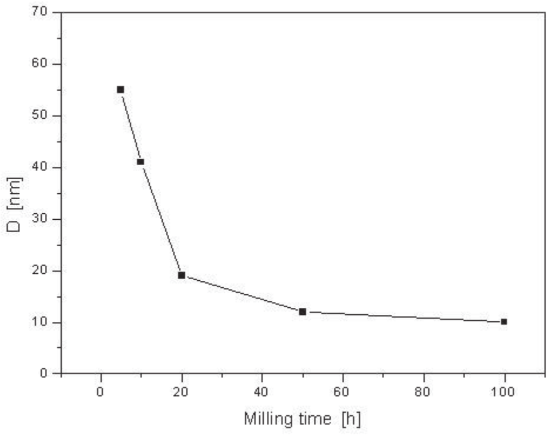

Fig. 4. The dependence of crystallite size of ferrite on milling time.

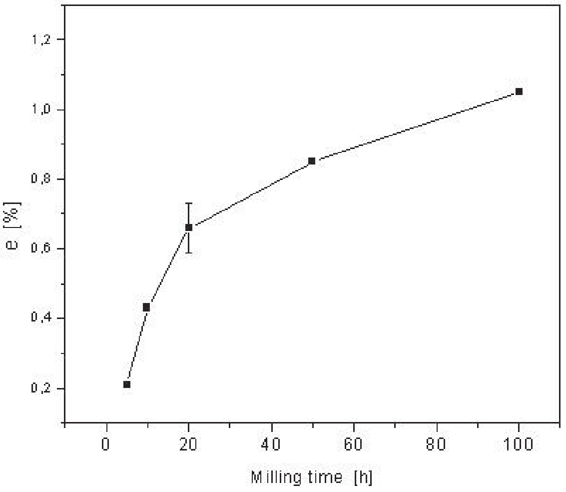

Fig. 5. The dependence of lattice strain in ferrite on mechanical alloying time.

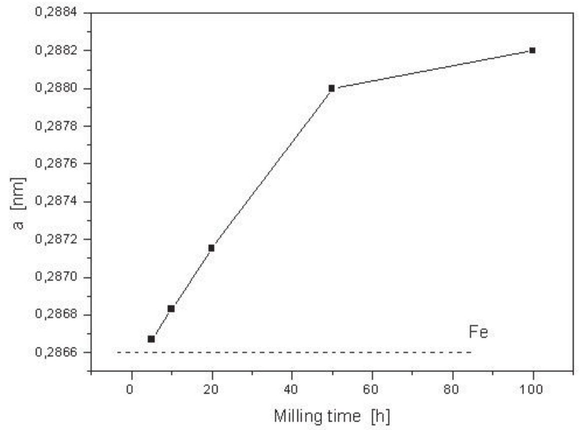

Fig. 6. The changes of lattice parameter of ferrite with milling time.

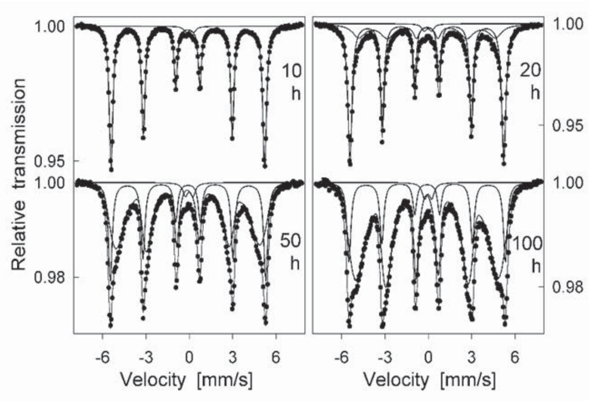

Fig. 7. The Mössbauer spectra recorded for the samples after different mechanical alloying times.

ences are small and also depend on the content of the alloying elements. Fe_3C from iron and graphite does not contribute significantly to the structure. The relative contents of the ferrite, alloyed ferrite and carbides after 100 h of mechanical alloying were calculated as 20, 71 and 9%, respectively. The results of Mössbauer analysis are collected in Table I. It is worthwhile to underline, that Mössbauer investigations allowed the detection of carbides in the mechanically alloyed samples, which were invisible at the standard XRD patterns.

The DSC curve registered during the heating of the powders mechanically alloyed for 100 h is shown in Fig. 8. Two exothermic effects are visible: a broad one in the temperature range 300-650 °C and a sharp one with a peak temperature at 700 °C. In order to explain these effects, the samples heated up to 650 and 720 °C were subjected to XRD studies. Fig. 9 shows the XRD patterns of the samples after 100 h of MA and after MA followed by heating up to 650 and 720 °C, i.e. above the first and second exothermic effects, respectively. After heating the MA powders up to 650 °C, only ferrite lines are observed, however much narrow than after MA only. Heating the sample to the higher temperature leads to the appearance of the carbide diffraction lines in the spectrum. This investigation allowed to found that the first exoeffect corresponds to the significant decrease of lattice strain down to 0.26% and simultaneously the increase of ferrite crystallite size is not observed. On the other hand, the high temperature DSC peak can be attributed to the carbide formation and growth, accompanied by the increase of ferrite crystallite size up to 35 nm.

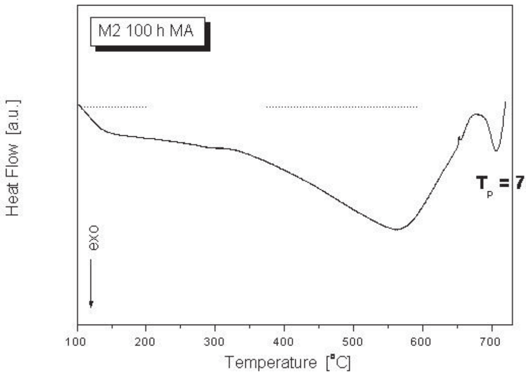

Fig. 8. DSC curve recorded during heating of the powders mechanically alloyed for 100 h.

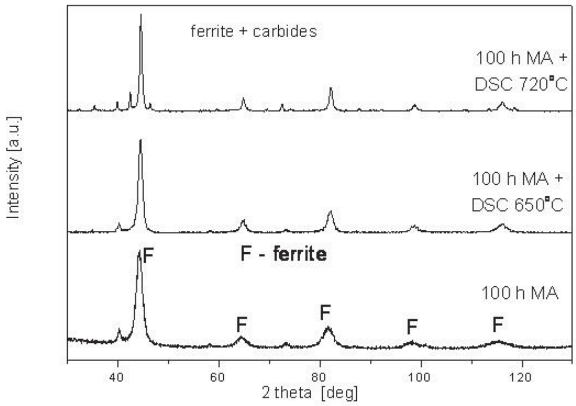

Fig. 9. XRD patterns of the samples subjected to heating in the DSC up to various temperatures.

Table 1. Hyperfine parameters (hyperfine field H_{hf}, isomer shift δ, quadrupole splitting Δ and relative content A) of the phases observed in the samples after mechanical alloying, determined from the Mössbauer spectra.

Milling time [h]	Ferrite			Ferrite alloyed			Carbides		
	H_{hf} [T]	δ [mm/s]	A [%]	H_{hf} [T]	δ [mm/s]	A [%]	Δ [mm/s]	δ [mm/s]	A [%]
2	33.0	0.00	98	-	-	-	0.53	0.10	2
5	33.0	0.00	98	-	-	-	0.46	0.09	2
10	33.0	0.00	98	-	-	-	0.51	0.14	2
20	33.1	0.00	71	29.4	0.02	27	0.52	0.07	2
50	33.4	0.01	33	30.0	-0.05	60	0.53	0.06	7
100	33.6	0.02	20	29.6	0.00	71	0.55	0.08	9

4. CONCLUSIONS

The mechanical alloying process of M2 high speed steel was performed using a mixture of pure elemental powders and Fritsch P5 planetary ball mill. The milling resulted in the formation of steel powders with average particle size about 4 μm and exhibiting round-in-shape morphology, typical for ball milled powders. The XRD studies revealed the formation of nanoferrite characterized by the final values (after 100 h of MA) of crystallite size and lattice strain of 12 nm and 1%, respectively. The Mössbauer measurements allowed to distinguish between ferrite and alloyed ferrite and to estimate the content of both phases after 100 h of MA as 20 and 71%, respectively. Moreover, the content of iron containing carbides was found as 9%, not revealed at the XRD patterns. During the heating in the DSC of the mechanically alloyed powders two exothermic effects were observed, corresponding to lattice strain decrease and carbide formation and growth, respectively.

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