A study of formation of iron nanoparticles in aluminium matrix with helium pores.

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Abstract. The structural and magnetic properties of iron nanoparticles formed in an aluminium matrix by combined helium and iron ions irradiation have been studied by small-angle X-ray scattering and susceptibility measurements. The formation of nanoparticles with average size of about 6.8 nm under iron ions irradiation has been found. The additional posterior irradiation with helium ions leads to the iron nanoparticles ordering with correlation length of 7.2(1) nm. The characteristic radius of iron nanoparticles and fractal dimension of the Al matrix were determined and magnetic properties of the Fe-Al nanomaterials were analysed.

1. Introduction

In recent years nanostructured materials have received increased attention due to their enhanced magnetic, mechanical and optical properties when the particle size is reduced to a few nanometers [1]. Improved magnetic properties like large coercitivity, giant magnetoresistance, superparamagnetism and high Curie temperatures are observed in nanostructured magnetic materials compared to their bulk counterparts [2]. These materials have significant potential for a wide range of applications such as catalysis, magnetic recording media, optoelectronics, magnetic fluids, fuel cells, sensors, etc. [3].

It is important to have tools for searching efficient and inexpensive techniques for production of nanostructured magnetic materials with control over composition, nanoparticles sizes and distribution. One of promising methods is the irradiation of materials by high energy charged particles [4]. This allows obtaining uniform volume concentration of the implanted ions in the matrix and provides possibility of doping any matrix by practically any element including chemically non-soluble in this matrix. Because the formation of nanoparticles is governed by fluctuation mechanism, one of the drawbacks of this method is the high-dispersed of nanoparticles formed [4]. Recently this was solved [5] by the additional pre-irradiation of the host matrix by inert gases ions forming the uniform in size
and spatially ordered pores in the matrix which can effectively trap the metal ions thus forming the monodispersed ordered nanoparticles [5]. The size and the density of nanopores can be controlled by varying the irradiation conditions such as a dose, intensity of the ions or temperature of the matrix.

The aluminum is a preferable material for the matrix due to its excellent corrosion and wear resistance, chemical stability. In the present work we focused on the study of structure aspects of iron nanoparticles formation in aluminum foils by helium and iron ions irradiation processes.

2. Experimental

The irradiations of aluminium matrix by helium and iron ions were carried out at ECR-1 cyclotron (Joint Institute for Nuclear Research, Flerov Laboratory of Nuclear Reactions, Dubna). The sample irradiation characteristics are presented in Table 1.

| Sample | The irradiation process characteristics | The corresponding ions energy and doses |
|--------|----------------------------------------|----------------------------------------|
| 1      | Helium ions only                        | E(He\(^{+}\)) = 20 keV, Dose = 5x10\(^{17}\) cm\(^{-2}\) |
| 2      | Helium ions and subsequent iron ions    | E(He\(^{+}\)) = 20 keV, Dose = 5x10\(^{17}\) cm\(^{-2}\) E(Fe\(^{+}\)) = 150 keV, Dose = 1x10\(^{15}\) cm\(^{-2}\) |
| 3      | Iron ions only                          | E(Fe\(^{+}\)) = 150 keV, Dose = 1x10\(^{15}\) cm\(^{-2}\) |
| 4      | Iron ions and subsequent helium ions    | E(He\(^{+}\)) = 20 keV, Dose = 5x10\(^{17}\) cm\(^{-2}\) E(Fe\(^{+}\)) = 150 keV, Dose = 1x10\(^{15}\) cm\(^{-2}\) |

The small-angle X-rays scattering (SAXS) experiments were carried out using the Structural Materials Science end-station [6] at the Siberia-2 synchrotron radiation source in RRC “Kurchatov Institute” (Moscow, Russia). The vacuum small-angle scattering chamber has a few sample loading ports at different distances to the detector for covering different scattering vector range. The minimum value of the accessible scattering vector is about 0.05 nm\(^{-1}\). The SAXS data were recorded using a one-dimensional gas-filled detector. In all spectra obtained, the background and detector anisotropy correction were carried out.

The magnetic DC susceptibility measurements were performed using a SQUID magnetometer. The temperature dependences of the susceptibility were measured in the magnetic field of 1 T. The magnetic field dependences of the susceptibility up to 5 T were measured at T = 5 K.

3. Results and discussion

The small-angle X-rays scattering curves of measured samples Nos. 1-4 (fig. 1) exhibit clear distinctions. For the fitting of the SAXS data we used structural model [7, 8] involving contribution from both Al matrix and nanoparticles formed inside the matrix:

\[
d\Sigma(q) / d\Omega = A q^{-\alpha} + B \exp(-\frac{1}{3} q^2 R_g^2) + D
\]

where the first term is due to fractal structure of the matrix with fractal dimension \(\alpha\). The second term is the Guinier term and depends on iron nanoparticles size \(R_g\). The \(B\) is Guinier prefactor which depends on number density of nanoparticles and \(A\), the scaling prefactor, signifies the strength of power-law scattering arising from the fractal structure [8, 9]. The calculated values of \(\alpha\) and \(R_g\) for different aluminium foils are listed in Table II.
Figure 1. The small angle X-ray scattering curves for irradiated aluminium foils. The solid lines are experimental data fit by equation (1) (curves 1-3) and (2) (curve 4). The “q_{or}” indicate the ordering peak for sample 4.

The scattering curve related to aluminium matrix irradiated by He^+ ions only (sample 1) corresponds to scattering from pure helium pores.

Table 2. The structural and magnetic properties of Fe nanoparticles in aluminium foils

| Sample | Gyration radius $R_g$, nm | Fractal dimension $\alpha$ | Bloch degree $b$ |
|--------|---------------------------|---------------------------|-----------------|
| Sample 1 | 6.95(1)                   | 2.43(2)                   | 1.27(4)         |
| Sample 2 | 10.4(1)                   | 1.94(1)                   | 1.62(3)         |
| Sample 3 | 7.10(2)                   | 2.10(3)                   | 1.58(3)         |
| Sample 4 | 6.80(2)                   | 2.12(4)                   | 1.58(2)         |

The application of Fe ions irradiation increases the total scattering intensity due a scattering length change [10] in the nanosystems formed. The SAXS curve corresponding to the sample 2 with aluminium matrix irradiated with helium ions followed by irradiation with iron ions is characterized by maximal scattering intensity. This corresponds to the maximal iron nanoparticles characteristic size (Table 1).

The SAXS curve of the sample 4 with aluminium matrix irradiated by iron ions with subsequent helium ions irradiation demonstrates the additional peak at $q=0.87$ nm$^{-1}$ (fig. 1). This feature can be
explained by iron nanoparticles ordering in aluminium matrix. For the fitting of this curve the modified equation (1) have been used:

\[
\frac{d\Sigma(q)}{d\Omega} = Aq^\alpha + B\exp(-\frac{1}{3}q^2R_g^2) + C\exp\left(-\frac{1}{\sigma}\right)\exp\left(-\frac{q-q_0}{\sigma}\right)
\]

(2),

where the third term describes the peak related to the ordering of Fe nanoparticles in Al matrix. The calculated values of peak width and position are \(\sigma = 0.05(2)\) nm\(^{-1}\) and \(q_0 = 0.87(2)\) nm\(^{-1}\). The corresponding correlation length for the ordered nanoparticles is \(D = 2\pi q_0 \approx 7.2(1)\) nm.

The magnetic susceptibility curves as functions of magnetic field and temperature are presented in fig. 2. The maximum \(\chi\) value at \(T = 300\) K corresponds to the sample 2, as one would expect due to larger characteristic size of iron nanoparticles (table 1).

![Graph](image)

**Figure 2.** The magnetic susceptibility curves as functions of magnetic field (a) fitted by liner function, and temperature (b), fitted by function (3) for the studied samples 1–4.

For the temperature range well below the Curie temperature, the temperature dependence of magnetization can fitted in frame of a spin-wave fluctuations theory by Bloch equation [11]:

\[
M = M_0(1 - BT^b)
\]

(3),

where \(B\) and \(b\) are the Bloch constants [11]. The obtained \(b\) values are listed in Table 2. For the samples 2, 3 and 4 they are close to ideal “Bloch degree” value \(b \approx 3/2\) and indicate the presence of small-sized magnetic nanoparticles [11]. The existence of small magnetization in sample 1 irradiated
by helium ions only can be explained by the presence of some impurities. This sample is characterized by the lowest Bloch degree $b=1.27(4)$.

**Conclusions**

The formation of iron nanoparticles in aluminium matrix by irradiation of iron and helium ions has been studied by means SAXS and magnetic measurements. The irradiation of Al matrix by He ions with a subsequent Fe ions irradiation leads to a formation of the disordered Fe nanoparticles array with the largest characteristic nanoparticle size of 10.4 nm. The opposite treatment, including irradiation of Al matrix by Fe ions with a subsequent He ions irradiation leads to a formation of the ordered Fe nanoparticles array with a correlation length of 7.2 nm and characteristic nanoparticle size of 6.8 nm.

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