



The study of magnetic properties and relaxation processes in Co/Au bimetallic nanoparticles



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ABSTRACT

Co/Au bimetallic fine nanoparticles were prepared employing the method of microemulsion using reverse micelle as nanoreactor, controlling the particles size. Magnetic and structural properties of two different samples Co/Au1 and Co/Au2 with almost comparable size of Co core and different size of Au layer were studied. The investigation of magnetic relaxation processes present in the particles was carried out by means of *ac* and *dc* magnetization data obtained at different temperatures and magnitudes of magnetic field. We observed the existence of superspin glass state characterized by the strong inter-particle interactions in the nanoparticle systems. In this paper, we discuss the attributes of novel superspin glass magnetic state reflected on various features (saturated FC magnetization at low temperatures, shift of the Cole–Cole arc downwards) and calculated parameters (relaxation time, critical exponent $z\nu \sim 10$ and frequency dependent criterion $p < 0.05$). Comparison of the magnetic properties of two studied samples show that the thickness of diamagnetic Au shell significantly influences the magnetic interactions and change the relaxation dynamics.

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1. Introduction

Magnetic nanoparticles have been investigated from different aspects for more than three decades. Its unique properties are suitable for numerous applications. Their introduction in medicine (cancer diagnosing [1], cancer treating [2], drug delivery [3]) or environmental science [4] posed new trends and challenges in these fields for the incoming 21st century. Therefore, the understanding of processes and phenomena present in the magnetic nanoparticle systems is crucial.

Unique properties of monodomain magnetic nanoparticles stem in existence of giant magnetic moment, superspin (several orders larger than the magnetic moment of single atom), in individual nanoparticle. The superspin can freely fluctuate along the magnetocrystalline axis [5]. If the relaxation time of superspin reversal is short, superparamagnetic state is recognized [5–9]. However, this behavior can be suppressed by the influence of temperature, external magnetic field as well as by the effect of magnetic inter-particle interactions. Proper combination of these conditions slows the magnetic moment relaxation and may cause superspin blocking, superspin freezing or even superspin long range ordering

in magnetic nanoparticles [5,6]. Several works concerning the influence of the strength of inter-particle magnetic interactions on magnetization relaxation have been reported [7–11]. These studies show that with increasing concentration of the particles in the assembly the dipole–dipole magnetic interactions between superspins became predominant and collective behavior appears. It is considered to be the origin of superspin glass state observed and discussed in the last years [9,10]. The concept of superspin glass state was introduced for the magnetic state of the nanoparticle assembly which shows magnetic characteristics similar to the atomic spin glasses. However, the origin of the interactions controlling the magnetic ordering is completely different in both cases. De Toro et al. [11,12] observed the ideal superspin glass behavior in the ensemble of uniform 8 nm maghemite nanoparticles after they had been pressed into the disc. On the other hand, Tadic et al. [13] presented non-compressed maghemite nanoparticles (4 nm) which exhibited superparamagnetic properties. These results are in accordance with the finding of Jimenez-Villacorta et al. [8] who were investigating the concentration effect on magnetic characteristics of the fine Fe nanoparticles embedded in non-magnetic matrix. They concluded the higher concentration of the particles affect the strength of inter-particle magnetic interaction and leads to the change of the superparamagnetic to superspin or even superferromagnetic state. Ebbing et al. [14] reported the influence

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of platinum capping on fine cobalt nanoparticles and documented the enhanced magnetic anisotropy and superspin glass behavior for the particles with thicker Pt layer. Song et al. [15] observed slight decrease of blocking temperatures with increasing coating layer in core@shell Co@Au nanoparticle system. They assumed the effect was induced by enhancing the inter-particle distances what led to the decay of the interaction strength. Corresponding with the theory of superparamagnetism, Su et al. [16] found the decrease of the blocking temperature with diminishing diameter of the Co nanoparticles. Also the values of coercivity of the Co nanoparticles measured below the blocking temperature turned out to be proportional to the volume of the particles. Obviously, superspin glass state can be induced by various factors.

Concerning the above mentioned facts we attempted to produce systems of cobalt/gold bimetallic nanoparticles with the aim to encounter the superspin glass state and to study the processes leading to its formation. In the presented paper we have studied magnetic state and relaxation process in cobalt nanoparticles coated by protective gold layer, prepared in two different samples with different size of gold shell. We have shown that the nanoparticles collectively behave like a superspin glass and exhibit different magnetic responses when the size of diamagnetic shell differs. The existence of superspin glass state in bimetallic Co/Au nanoparticles has not yet been published.

2. Experimental

2.1. Preparation

All chemicals, namely: Chloroauric acid (HAuCl_4 , 99.9%), Cobalt chloride (CoCl_2 , 97%) octane, 1-butanol (99.8%),

Cetyltrimethylammonium bromide (CTAB, 99%), sodium borohydride (NaBH_4 , 98%), were purchased from Aldrich and Sigma. Nanoparticles were prepared using microemulsion method in reverse micelles [17]. All reverse micelle solutions were prepared using CTAB as the surfactant with octane as the oil phase. 1-butanol was used as co-surfactant, increasing the polarity of the surfactant and helping to stabilize the micelle solutions [17]. Aqueous reactants of CoCl_2 , NaBH_4 , and HAuCl_4 were used to form the reverse micelle. The metal particles were formed inside the reverse micelle by reduction of a metal salt using sodium borohydride. At first the pure Co core were produced using a magnetic stirrer and under flowing argon. Co based nanoparticles were subsequently coated by Au shell also by using of reverse micelle solutions. After nanoparticle preparation, the micelles in the reaction mixture were disrupted using acetone causing nanoparticles to precipitate. Repeated washing using a 1:1 mixture of chloroform/methanol removed the surfactant. The size of prepared nanoparticles was controlled by the water to surfactant molar ratio w ($[\text{H}_2\text{O}]/[\text{CTAB}]$). Two different samples of Co/Au1 and Co/Au2 were prepared, where the same molar ratio for preparation of Co core ($w_{\text{Co}} = 8$) and different ratio for Au shell ($w_{\text{Au1}} = 5$ and $w_{\text{Au2}} = 10$) were used.

2.2. Characterization

The high-energy powder X-ray diffraction (HE-PXRD) experiments were carried out at BW5 wiggler beamline of DORIS positron storage ring in DESY (Hamburg, Germany) using monochromatic synchrotron radiation with beam energy of 100 keV ($\lambda = 0.12398 \text{ \AA}$). The experiments were carried out at room temperature in the transmission mode. LaB_6 standard was used to

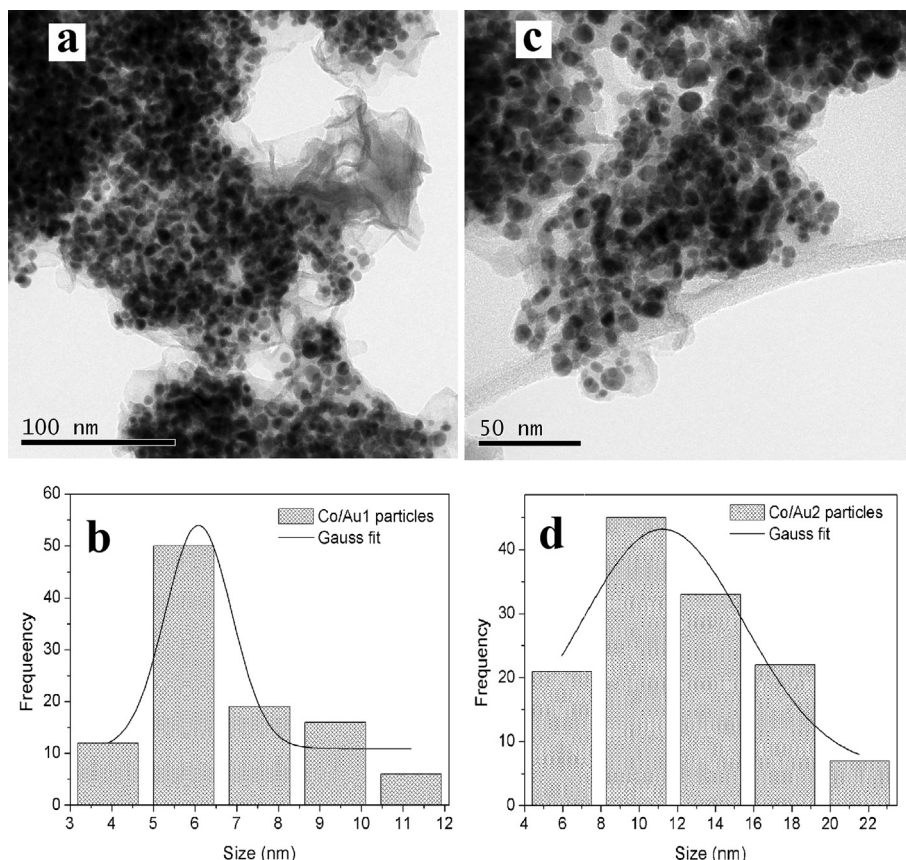


Fig. 1. High resolution transmission electron images of samples Co/Au1 (a) and Co/Au2 (c) samples and its size distributions (b), (d), respectively.

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