

Magnetic anisotropy effects in nano-cluster nickel particles

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Abstract

Nano-cluster Ni particles with average particle size from 12–100 nm were successfully prepared by the evaporation technique. For samples with an average particle size below roughly 50 nm, we observed a nonsymmetric magnetic hysteresis loop and a deviation between the zero field cooling (ZFC) and the field cooling (FC) magnetizations at temperatures below a critical temperature T_x . T_x is a function of the applied magnetic field and the size of the nickel particles which is in general inversely proportional to the saturation magnetization of the nickel particles. This can be explained by the effect of exchange anisotropy interaction between the interfaces of the ferromagnetic region of Ni nano-clusters and the layers of antiferromagnetic NiO on the surface of the nano-cluster Ni particles. A slope variation in magnetization below a special temperature T_s (roughly 50 K) for nano-cluster Ni particles with average particle sizes roughly below 50 nm has been observed. This is explained by a spin flip behaviour near the interfaces between the nano-cluster Ni particles and the layers of NiO on the surface of the Ni particles.

Keywords: Magnetic anisotropy; Nano-clusters; Nickel

1. Introduction

In recent year research on nano-cluster magnetic particles has been very active because of the wide range of potential applications; including high density magnetic recording media, ceramics, catalysts, drug delivery systems, ferrofluids, pigments in paints and medical diagnostics [1,2]. Nano-cluster-sized crystalline materials are polycrystals in which the size of the individual crystallites is of the order of several nanometres. Therefore, a large fraction of the atoms in nano-cluster particles are surface or interface atoms. Any structure sensitive physical properties are expected to differ between nanocluster particles and bulk materials.

In this investigation, we report on the study of the magnetic anisotropy effect in nano-cluster nickel particles as a function of particle size, applied magnetic field, and temperature.

2. Experimental

Samples for this study were prepared from commercially available high purity Ni ingots (99.999%) and NiO (99.995%) powders. Nanocrystalline Ni powders

with an average particle size from 12–100 nm were prepared by the evaporation technique. The pressure of the He gas atmosphere during evaporation was varied from 1–100 mbar to control the growth of the particle size. The magnetization below 300 K was studied by using both superconducting quantum interference device (SQUID) and vibrating sample magnetometer (VSM). Powder X-ray diffraction and transmission electron microscopy (TEM) were used in structure, morphology and particle size analyses.

3. Results and discussion

TEM was used to study the crystal structure and particle morphology. Fig. 1(a) shows, as an example, the TEM micrograph for nano-cluster Ni particles with an average particle size around 22 nm. It is clear that nearly spherical particles with negligible shape anisotropy and with strong tendency of forming chains among the particles were obtained. The particle size distribution was obtained by measuring about 500 Ni particles from TEM micrographs. X-ray diffraction patterns of the nano-cluster Ni particles (22 nm), the NiO powder particles (1000 nm), and the Ni bulk

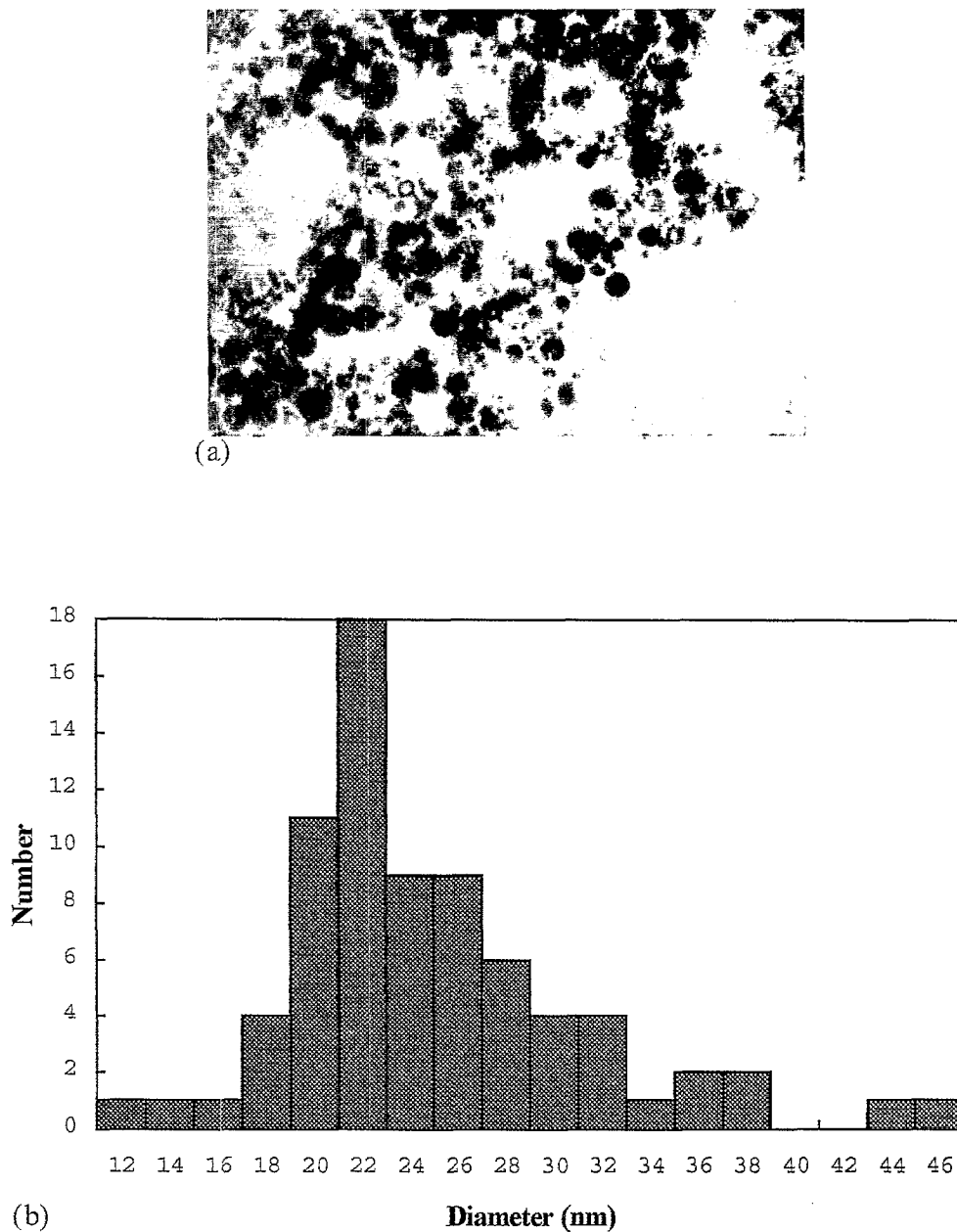


Fig. 1. (a) TEM micrograph and (b) particle size distribution for nano-cluster Ni particles with average particle size around 22 nm.

samples have been reported before [3]. The main peaks of the X-ray diffraction pattern of the nano-cluster Ni particles are completely consistent with those of the pure Ni phase, except that the peaks become more broad. From this X-ray study, we can infer that the concentration of Ni oxide on the surface of nano-cluster Ni particles should be less than a few percent. This is quite similar to the nano-cluster Cu and Fe particles reported before [4–6].

The magnetic exchange anisotropy effect in nano-cluster Ni particles has been partially studied before [3], and it was not clear, at that time, whether to distinguish the critical temperature T_x (T_x is defined as the temper-

ature below which both a shifted magnetic hysteresis loop and a deviation in the magnetization between the zero field cooling (ZFC) and the field cooling (FC) occur) and the special temperature T_s (T_s is defined as the temperature below which a faster increasing of the magnetization with decreasing temperature occur). We know that the magnetic exchange anisotropy effect is due to the interaction between the interfaces of the ferromagnetic core region and the layers of antiferromagnetic NiO on the surface of the nano-cluster Ni particles. In this study, we find that, for nano-cluster Ni particles with an average particle size below 50 nm, T_x could be varied as a function of the applied magnetic

field and the size of the nano-cluster Ni particles which is coated by its oxide. However, T_s seems only observed in the FC case and is always closed to around 50 K. For example, Fig. 2 shows the FC and ZFC curves of the magnetization of nano-cluster (22 nm) Ni particles as a function of temperature with the applied field of 8 kOe. The two curves start to deviate from each other below roughly 50 K. However, for the same sample when the applied field is kept at 2 kOe, the deviation temperature of the two curves, as shown in Fig. 3, is roughly below 90 K. The magnetic hysteresis loops for the applied magnetic field at 8 kOe has been reported before [3]. It showed that the loops are nonsymmetrical for temperatures roughly below 50 K and are symmetrical for temperatures roughly above 50 K. However, the magnetic hysteresis loops for an applied magnetic field at 2 kOe are non-symmetrical for temperatures roughly below 90 K as presented in Fig. 4. The field dependence of the T_x is shown in Fig. 5. This could be understood, if we think that there is an infinite number of minor hysteresis loops inside the major loop and it is usually more asymmetrical than the major loop; that means the T_x is higher when the applied field is lower.

As shown in Fig. 5, the T_s , measured within our experimental error limitation, is always near 50 K for all the applied field below 8 kOe and for nano-cluster Ni samples with an average particle size below 50 nm. This effect disappears when the nano-cluster Ni samples are annealed at 500 °C in vacuum for 24 h, this means that the nano-cluster samples aggregate to a bulk Ni.

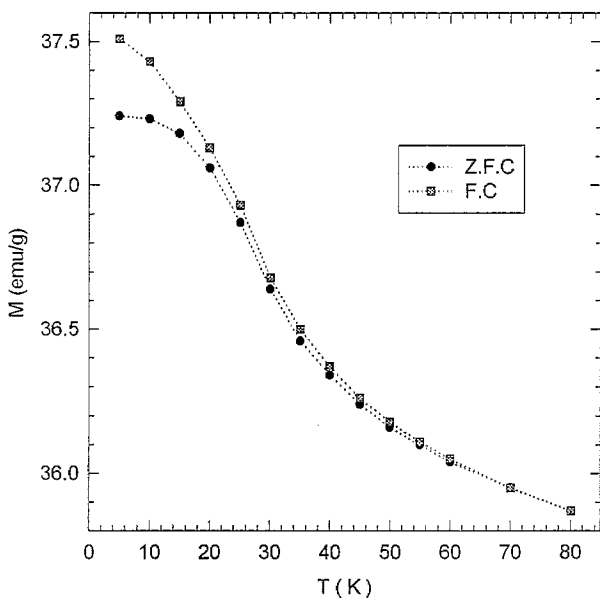


Fig. 2. ZFC and FC magnetization curves with applied field at 8 kOe as functions of temperature between 4–300 K for nano-cluster (22 nm) Ni particles.

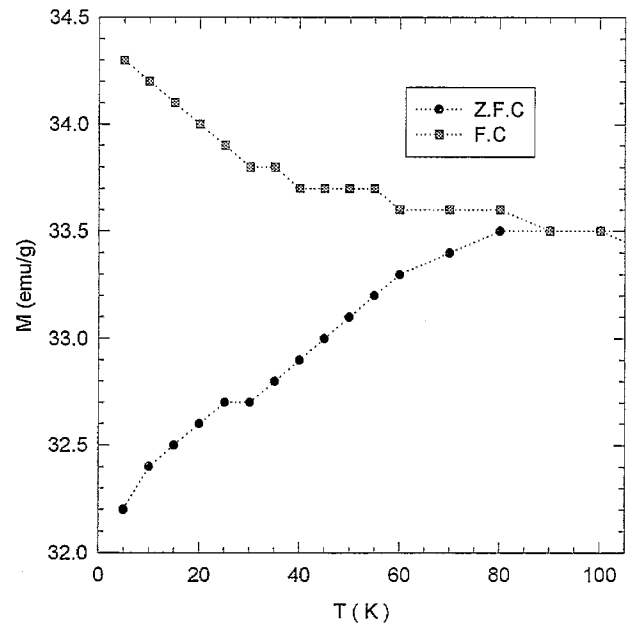


Fig. 3. ZFC and FC magnetization curves with applied field at 2 kOe as functions of temperature between 4–300 K for nano-cluster (22 nm) Ni particles.

If the nano-cluster Ni particles are annealed at 500 °C in air, its surface quickly oxide. Its magnetization decreases as a function of annealing time. As an example, Fig. 6 shows $M(T)$ for the nano-cluster (22 nm) Ni samples annealed at 500 °C in air for 60 min. The two curves start to deviate below roughly 300 K. The magnetic hysteresis loops for the same sample of Fig. 6 are plotted in Fig. 7. With different oxidation

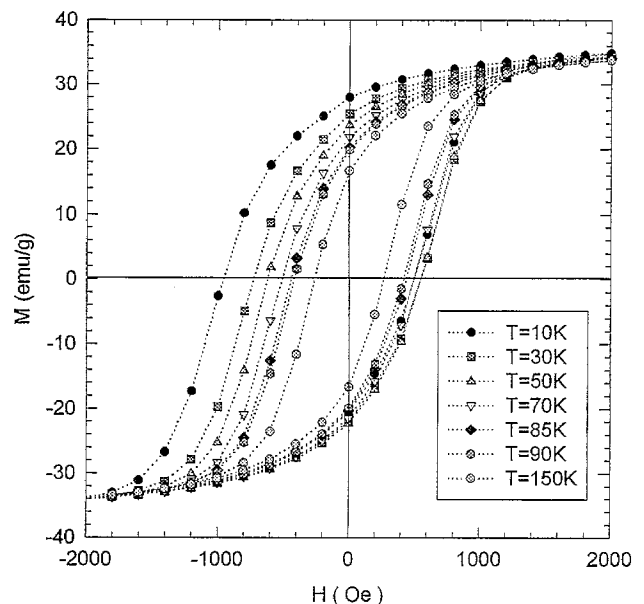


Fig. 4. FC magnetization vs applied field below 2 kOe at $T = 10, 30, 50, 70, 85, 90,$ and 150 K for nano-cluster (22 nm) Ni particles.

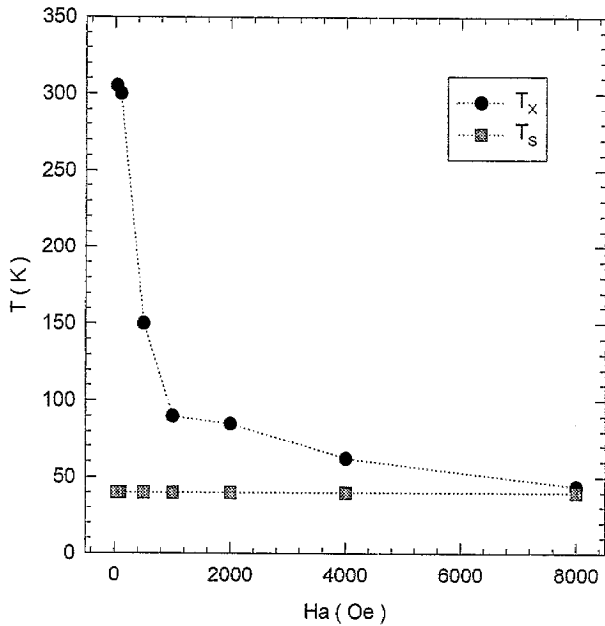


Fig. 5. T_x and T_s as functions of the applied field for nano-cluster (22 nm) particles.

condition, we can obtain all the points of T_x . However, for the oxidized nanocluster Ni particles, we can not obtain the accurate size of the nano-cluster Ni particle. The only meaningful physical quantity is the magnetization and its value should be proportional to the size of the nano-cluster Ni core. Therefore, we plotted the variation of T_x and T_s obtained

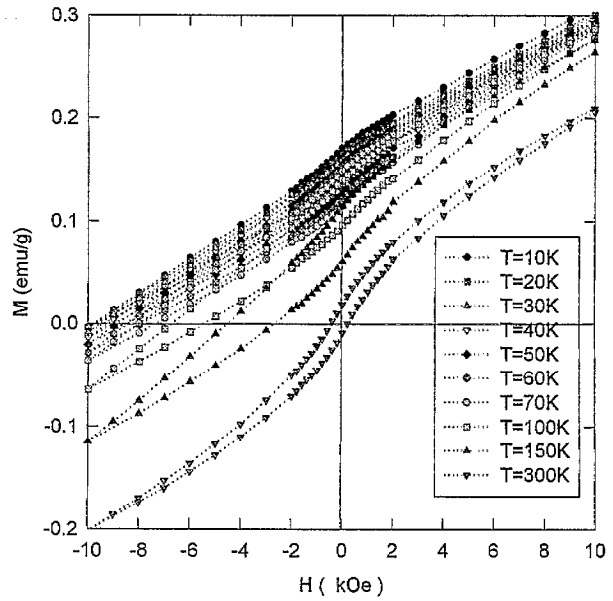


Fig. 7. FC magnetization vs applied field below 10 kOe at $T = 10, 20, 30, 40, 50, 60, 70, 100, 150,$ and 300 K for oxidated nano-cluster (22 nm) Ni particles.

experimentally as functions of the saturation magnetization for all the oxidated samples in Fig. 8. Again T_s is around 50 K within the experimental error limitation, and T_x seems to vary inversely proportional to the saturation magnetization. This suggests that it is easier to get magnetic anisotropy for the smaller metallic Ni core in an oxidated nano-cluster Ni particle.

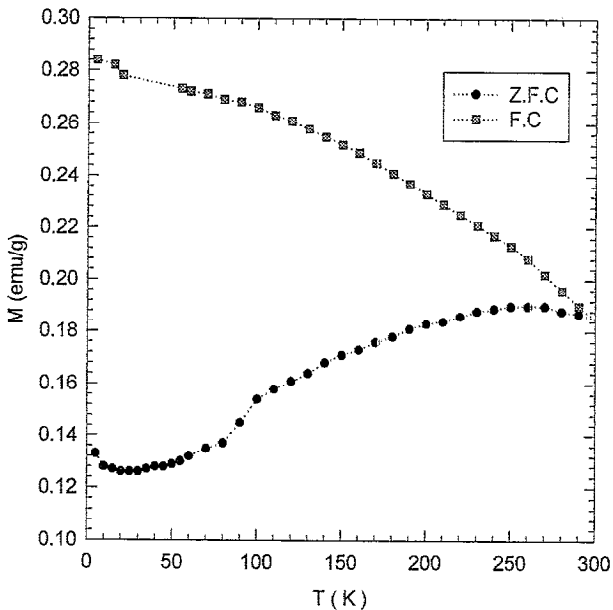


Fig. 6. ZFC and FC magnetization curves with applied field at 8 kOe as functions of temperature between 4–300 K for oxidated nano-cluster (22 nm) Ni particles.

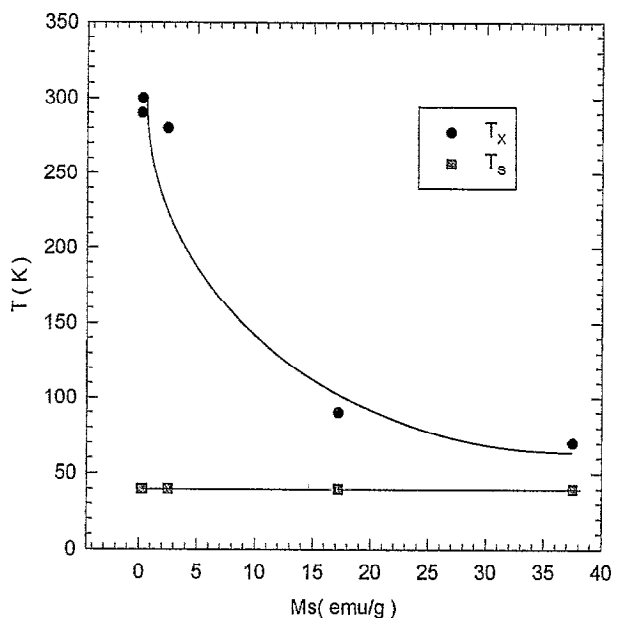


Fig. 8. T_x and T_s as functions of the saturation magnetization for oxidated nano-cluster (22 nm) particles.

In conclusion, we have reported the magnetic anisotropy effect in nano-cluster Ni particles. We observed that the nonsymmetric behaviour of the magnetic hysteresis loop is strongly related to T_x . T_x varies as a function of the applied field and the size of the metal core of the oxidated nano-cluster nickel particles which is assumed to vary inversely proportional to its saturation magnetization. However, the temperature T_s below which a faster increase of the magnetization with decreasing temperature occurs, seems always very close to 50 K for all the nano-cluster Ni particles with average particle size roughly below 50 nm. These phenomena have been qualitatively explained by the exchange anisotropy and the spin flip behaviours near the interface of the nano-cluster Ni particles and their oxide layer on the surface. A detailed quantitative analysis shall be reported elsewhere.

Acknowledgements

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References

- [1] M. Ozaki, *Mater. Res. Bull.* XIV, (1989) 35.
- [2] H. Gleiter, *Nanostruct. Mater.*, 1 (1992) 1.
- [3] Y.D. Yao, Y.Y. Chen, C.M. Hsu, H.M. Lin, C.Y. Tung, M.F. Tai, D.H. Wang, K.T. Wu, and C.T. Suo, *NanoStruct. Mater.*, 6 (1995) 933.
- [4] Y.Y. Chen, Y.D. Yao, B.T. Lin, S.G. Shyu, and H.M. Lin, *Chin. J. Phys.*, 32 (1994) 479.
- [5] S.U. Jen, C.Y. Lee and Y.D. Yao, *J. Magn. Magn. Mater.*, 96 (1991) 82.
- [6] C.Y. Lin, K.C. Lee and Y.D. Yao, *Solid St. Comm.*, 83 (1992) 371.