Ferromagnetic fraction and exchange anisotropy in SUS 316LN austenitic stainless steel due to strain-induced deformation

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The exchange bias behavior was observed between strain-induced ferromagnetic (FM) α' martensite and antiferromagnetic (AFM) austenitic phase in SUS 316LN stainless steel at <30 K. Based on the magnetic hysteresis loops, the exchange contact was evident when a small amount of FM was formed, typically about <1.0 vol %. The exchange bias field (H_{ex}) was reported here as a function of FM volume fraction at different temperatures. The decreasing H_{ex} with FM is attributed to the increasing size of FM crystallites in the AFM matrix. © 2008 American Institute of Physics. [DOI: 10.1063/1.2830831]

I. INTRODUCTION

SUS 316LN is a molybdenum bearing nitrogen-alloyed low-carbon austenitic steel. It is paramagnetic at room temperature and antiferromagnetic (AFM) below its blocking temperature (T_b). Type 316LN alloy is used in widespread industrial and technological applications ranging from nuclear reactors to cryogenic equipments because of its superior mechanical and chemical properties. It is a candidate material for fusion reactors wherein the material must remain nonmagnetic to avoid interactions with the magnetic field produced by the superconducting magnets.^{1,2} Hence, it is important to investigate the magnetic behavior of this material especially in the cryogenic temperatures.

It is well known that strain-induced deformation can transform the austenitic structure (fcc, γ) to ferromagnetic (FM) α' martensite (bct) in stainless steels, which depends on the chemical composition of the alloy, temperature, strain rate, etc. In type 316LN steel, a relatively stable alloy among 300 grade steels, such a $\gamma \rightarrow \alpha'$ transformation was considered to be a hindered process when deformed at room temperature, unlike the low-temperature deformations.^{1,2}

Exchange bias refers to the effect where the magnetization of a FM layer is pinned into a well-defined reference direction by an AFM such that its hysteresis loop exhibits a horizontal shift when field cooled from above T_b . Conventionally, exchange interaction was observed due to contact interface between the FM and AFM layer systems such as thin films, fine particles, and nanoparticles of FM core covered with AFM shell.^{3,4} However, such an exchange interaction is not commonly seen in the crystalline alloys which was first reported on cold-worked-type 347 stainless steel by Meiklejohn.⁴

Although few studies have mentioned such exchange interaction between the strain-induced FM and austenitic phase in stainless steels,^{5,6} there is no detailed information on the extent of such exchange contact as a function of α' martensite volume fraction. In summary, the possible AFM/FM in-

teractions in austenitic steels have received less attention. In this study, we have investigated this aspect using SUS 316LN alloy which was compressively deformed at room temperature (RT) and also at liquid-N₂ temperature.

II. EXPERIMENTAL PROCEDURE

The chemical composition of the SUS 316LN alloy is shown in Table I. The samples machined to $3 \times 3 \times 2$ mm³ were solution annealed at 1050 °C for 80 min, water quenched and thereafter electropolished and cleaned. Then, compressively deformed at RT at a strain rate of 0.5 mm/min and obtained different amounts of FM (<0.2 vol %) corresponding to different true strains (ϵ_t). To achieve higher amount of FM, a few samples were compressed at 77 K ($\epsilon_t=30\% - 50\%$) wherein about 1–5 vol % FM was obtained. Magnetization was measured between 2 and 300 K using a superconducting quantum interference device magnetometer. The field applied (H) was perpendicular to the axis of compressive deformation.

During the hysteresis (M - H) loop measurement, the applied field was cycled from 30 to -30 kOe. The exchange contact was observed in the field-cooled (FC) condition wherein the sample was cooled from 300 K to different low temperatures at $H_{\text{FC}}=30$ kOe. In zero-field-cooled (ZFC) condition, the sample was cooled from 300 to 2 K at $H=0$ and then rose to different temperatures.

Exchange bias field (H_{ex})= $(H_{c_L}-H_{c_R})/2$ where H_{c_L} and H_{c_R} are the left and the right intercepts of the M - H loop with the field axis. In this study, H_{ex} was obtained after subtract-

TABLE I. Chemical composition of SUS 316LN (wt %).

C	Si	Mn	P	S	Ni
0.015	0.58	1.39	0.022	<0.0005	12.71
Cr	Mo	N	Co	Cu	Fe
17.29	2.52	0.112	0.04	0.08	Bal.

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FIG. 1. (Color online) Magnetization of the undeformed sample indicating the paramagnetic and antiferromagnetic behaviors as a function of temperature.

ing the magnetization of austenite matrix (equivalent to the magnetization of the undeformed sample at the same condition that used for deformed samples).

III. RESULTS AND DISCUSSION

The blocking temperature (T_b) of SUS 316LN was determined to be ≈ 30 K as indicated from the magnetization plots of undeformed sample as a function of temperature under ZFC and FC conditions (Fig. 1). Thus, the alloy is paramagnetic above 30 K and below which it is AFM.

As shown in Fig. 2, the amount of FM α' martensite increased sharply after about 30% true strain as determined from the saturation magnetization (M_s) measured at RT. In these steels, austenite to α' martensite transformation is believed to follow^{7,8} as $\gamma(\text{fcc}) \rightarrow \text{stacking faults} \rightarrow \epsilon(\text{hcp}) \rightarrow \alpha'(\text{bcc})$. Further, it is known that α' -martensite grains ap-

FIG. 2. (Color online) Volume fraction of strain-induced α' martensite (FM) due to compressive deformation at RT. The inset shows the saturation magnetization plots measured at RT and after subtracting the magnetization of the austenitic matrix.

FIG. 3. (Color online) H_{ex} as a function of FM volume fraction at 5 K. The top inset is the enlarged view of the M - H loops, and the bottom inset is the H_{ex} vs T for the sample deformed to $\epsilon_t = 48.4\%$.

pear inside the austenite grains mainly at the intersection of shear bands, making the movement of dislocations more difficult.

The FM is presumed to be distributed homogeneously in the austenite matrix at this small amounts (< 0.2 vol %). When hysteresis loops were measured for such coexisting system, the symmetrical loop under ZFC became asymmetrical if field cooled through T_b . In a typical case as shown in the inset (top) of Fig. 3, it is apparent that the loop is shifted in the horizontally negative direction. The origin of this shift along the field axis is ascribed to the exchange contact at the boundary or interface of α' martensite in austenitic matrix,⁴⁻⁶ i.e., the microscopic torque is exerted by the AFM spins to the spins in the FM while reversing the magnetization of FM.

Since H_{ex} depends on the cooling field (H_{FC}), in this study, it was determined to be about 30 kOe as the H_{FC} and remanence reached a steady state.

Figure 3 shows H_{ex} as a function of FM volume fraction at 5 K, as a typical case. It is found that H_{ex} decrease from about 143 to 10 Oe as the FM was increased to about 1 vol % from a negligibly small amount. A similar behavior was observed at all the temperatures from 2 to 30 K, and thereafter the M - H loops were symmetrical and hence H_{ex} was zero. For comparison of the H_{ex} as a function of FM volume fraction, in Fig. 3, H_{ex} of the samples deformed at RT ($\text{FM} < 0.2$ vol %) are extrapolated with the sample deformed at 77 K ($\text{FM} \approx 1.0$ vol %) even though the microstructure of FM was expected to be different.¹

The bottom inset in Fig. 3 shows H_{ex} as a function of temperature for the sample deformed to 48.4% true strain. It is found that H_{ex} decreases from 50 to 10 Oe as the temperature approaches 30 K (the so-called T_b) from 5 K. This behavior is in corroboration with Fig. 1, and verifies that the exchange bias phenomenon in SUS 316LN system exists for $T < T_b$. This decreasing trend of H_{ex} with increasing temperature remained same for all other samples deformed to different true strains ($\text{FM} < 1$ vol %). The temperature dependent

feature of the exchange coupling can be qualitatively explained based on the thermal fluctuation of the magnetic moments.

It is clear from Fig. 3 that the exchange contact was established only when small volume fraction of FM was formed in austenitic structure, typically about <1.0 vol %, and beyond which H_{ex} was found to vanish.

The variation of H_{ex} with increasing FM amount may be explained based on the increasing size of α' martensite in AFM austenitic matrix. The α' -martensite crystallites, once nucleated, are distributed wide apart in the austenite matrix establishing intimate contact with AFM phase. Thus, in the initial stage, the ratio of interface area between FM sites and AFM matrix is higher and hence the higher exchange contact. Due to small deformation ($\epsilon_t < 30\%$), it seems that the number density of isolate α' martensite increase while the size remain more or less same. On further deformation ($\epsilon_t > 30\%$), the size of FM sites was expected to increase. Thus, the relative interface area decreased, consequently, H_{ex} decreased with increasing FM. Nevertheless, the detailed mechanism regarding how the microstructure can affect the H_{ex} is not clearly understood yet. For layered structures, competing models for the origin of H_{ex} have been proposed: “spin flop” model assumes a 90° angle between the magnetization of FM and AFM,⁹ and coupling by uncompensated moments at steps or grain boundaries.¹⁰ Further, as the exchange interaction seems to derive from the interface between FM and AFM components, it was regarded that interface disorder such as roughness, crystallinity, grain size, and interface impurity, could play an important role on the extent of exchange coupling.¹¹ The martensite cluster size at this small amounts (<1 vol %) is presumed to be of the order of

nanometer scale; however, any experimental evidence on the microstructure of α' martensite could not be obtained (using transmission electron micrography and X-ray diffraction) at this small volume fraction,¹² and also due to high dislocation density caused by deformation.

In summary, this study has clearly shown that the exchange anisotropy can be observed only when small amount of α' martensite (less than about 1.0 vol %) was formed in the austenitic steel, SUS 316LN. As shown in Fig. 3, H_{ex} decreased with increasing amount of FM, and increased as the temperature was lowered below T_b . The dependence of H_{ex} with the volume fraction of FM is attributed to the increasing size of FM sites in the surrounding AFM austenite matrix.

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