THE RELATTON BETWEEN THE TEMPERING EFFECTS AND THE INTRINSIC COERCIVITY OF SINTERED $SmCo_5$ ALLOY AT 750℃ ①

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ABSTRACT

The relation between the tempering effects of sinterd $SmCo_5$ alloy at 750 ℃ and its intrinsic coercivity (i^{Hc}) has been studied by the use of photoelectron energy spectrum, X−ray diffraction and high−voltage electron microscope. The result is that the cause of i^{Hc} dropping seriously for sintered $SmCo_5$ alloy tempered at 750 ℃ is not the eutectoid decomposition of $SmCo_5$ and the increase of oxygen. In fact, i^{Hc}dropping is caused by that some defect-rich regions in Sm_2Co_{17} decomposed form $SmC0_5$ from nucleation centers in reversed magnetization course.

Key wrods: sintered alloy, tempering effect, intrinsic coercivity, $SmCo_5$

1 INTRODUCTION

Wenstendorp found the tempering effect of sintered $SmCo_5$ alloy at 750 ℃ [1, 2]. The most important phenomenon of this effect is that the intrinsic coercivity of $SmCo_5$ Keeps on droping from 25 ℃ to 750 ℃. In 1972, Den Broeder pointed out that $SmCo_5$ decomposed into Sm_2Co_7 and Sm_2Co_{17} after tempered at 750 ℃[3]. In 1972−1973, Broeder and Smeggile believed that i^{Hc} of this alloy dropping at 750 ℃ was caused by the eutectoid decomposition of $SmCo_5$($SmCo_5 \rightarrow SmCo_7 + SmCo_{17}$.[4]). In 1976, Broeder proposed that i^{Hc} dropping at 750 ℃ resulted from more oxygen dissolving into $SmCo_5$. He was sure that oxygen increasing and Sm_2O_3 forming at 750 ℃ not only decreased the pinning on domain walls, but also promoted the formation of reversed magnetization nucleus and thus resulted in the dropping of i^{Hc} of $SmCo_5$[5]. In 1980, Fidleret pointed out that this effect related to the growing of a few 2:17 phases[6].

We made a systematic study on the relation between the tempering effect of sintered $SmCo_5$ and its intrinsic coercivity at 750 ℃.

2 EXPERIMENTAL PROCEDURE

2.1 *Sample Preparation*

Put 99.8% Sm and 99.5% Co into a non-self-consuming furnace (Ar protected). Melt them and obtain casting alloys. Crush them to 5 μm size in organic medium and shape them

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in 1.5 T magnetic field. The blank is sintered at 1,135 ℃ for 30 min, and then held at 900 ℃ and quenched to room temperature for 1 h.

2.2 *The Photoelectron Energy Spectrum of* $SmCo_5$ *alloy*

the prepared $SmCo_5$ sample was measured with AES at the condition of incident-ray energy 3 keV, current 1μA, testing voltage 6 eV, multiplying voltage 12,000 V, time constant 0.03 s, magnifying times 40, vacuum degree $2-4\times10^{-7}$Pa. The measurement condition of XPS is: the light source is Mg-Pd radiation (voltage is 8 kV, current is 30 mA), experiment voltage is 50 eV.

Fig.1 gives the measuring results of the distribution of Sm, Co in $SmCo_5$ from 25 ℃ to 900 ℃, from which we find no oxygen peak presents at 750 ℃.

Fig.2 presents the Photoelectron energy spectrum result for $SmCo_5$ tempered at 750 ℃. We find no oxygen peak appears neither.

2.3 *X-ray Diffraction Analysis*

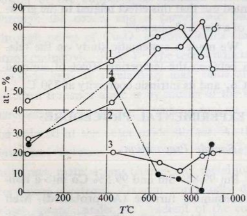

Fig.1 The distribution of Sm, Co, O of $SmCo_5$ tempered at temperature ***T***

1-Sm(no splashed); 2-Sm(Ar^+ splashed for 5 s);
3-O(Ar^+ splashed for 5 s); 4-Co(Ar^+ splashed for 5 s)

The result of X-ray diffraction shows that there exists only $SmCo_5$ phase at room temperature; but after tempered for 1 h at 420 ℃, Sm_2Co_7 and Sm_2Co_{17} phases appear. It means that part of $SmCo_5$ decomposes to eutectoid products Sm_2Co_7 and Sm_2Co_{17}.

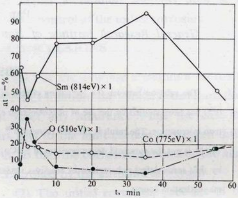

Fig.2 The distribution of Sm, Co, O of $SmCo_5$ tempered at 750 ℃ for 1 h

2.4 *Dynamic Observation on Samples With Transmissive Electron Microscope*

The preparation of the sample films used on elecron microscope is: First cut the sample into 0.25 mm thick films along the direction perpendicular to the easy magnetized axis, then cut these films into 100 nm thick with ion thinning machine. These samples are set onto TEM-1000 and heated on the conditions of 1000 kV acceleratiog voltage and 2.66×10^{-5} Pa vacuum, and then dynamic observation was carried out. The result shows that no precipitation appears before 350 ℃ but at 350 ℃ small precipitation can be found, and at 420 ℃, the field of vision is full of high-spread precipitations at the former place where there was nothing. Being kept at this temperature for 10 min the precipitation grow up to 10 nm and some gathered. This kind of precipitation is regarded Sm_2Co_{17} with electron diffraction analysis. There are two sets of diffraction patterns in the precipitation, which are regarded as Sm_2Co_7 and Sm_2Co_{17} phases. The defects in Sm_2Co_{17} phase can be seen from 600 ℃ to 800 ℃ and are especially obvious at 750 ℃.

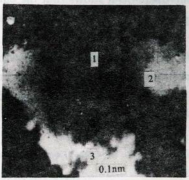

Fig.3 The defects (1, 2, 3) in Sm_2Co_{17} phase

Besides defects, some stripes can also be observed in Sm_2Co_5 at 750 ℃. Whether these stripes appearing after the $SmCo_5$ was tempered at 750 ℃ for 8 min indicate a new phase remains to be determined.

2.5 *The Variation of Intrinsic Coercivity With Tempering Temperature*

The thermal-demagnetized samples were tempered at 250, 350, 420, 500, 600, 700, 850, 900 and 1,000 ℃ respectively for 1h. The measurement results are shown in Fig.4. The intrinsic coercivity increases with temperature and its variation is nonlinear. At 750 ℃, coercivity reaches a minimum.

3 DISCUSSION

(1) After tempered at 420 ℃ for 20 min, part of $SmCo_5$ phase decomposes to Sm_2Co_7 and Sm_2Co_{17}. From Fig.4 we found no decreasement of the intrinsic coercivity of $SmCo_5$ at 420 ℃ comparing to that at room temperature. So we draw a conclusion that the eutectoid decomposition of $SmCo_5$ at 750 ℃ is not the reason for i^{Hc} dropping.

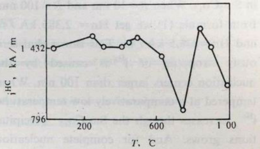

Fig.4 The Intrinsic coercivity(i^{Hc}) of $SmCo_5$ tempered at diffferent time(*T*) for 1 h

(2) The X-ray major diffraction spectrum shows that part of $SmCo_5$ tempered at 420 ℃ transfers into Sm_2Co_7 and Sm_2Co_{17}.

(3) The photoelectron energy spectrum shows that no oxygen peak appears at 750 ℃. So it is not proper to regard oxygen gathering as the reason for i^{Hc} of $SmCo_5$ dropping at 750 ℃.

(4) With an electron microscope, the precipitated Sm_2Co_{17} phase continues growing up and do the defects in it. Some stripes appear if the sample was tempered at 750 ℃ for 80 min, which remain to be studied. The segregation of Sm and Co caused by temperature variation result in the defects in Sm_2Co_{17} phase. The defects become reversed magnetization nucleation centers and make $i^{Hc}$of the $SmCo_5$ dropping[17].

(5) The reason for i^{Hc} dropping at 750℃ is the creation and growing of the reversed magnetization nucleation centers of each $SmCo_5$ grain. The size of these centers is fairly large and inside magnetic anisotropy is very small. If the radius(*R*) of reversed magnetization nucleus is constant, the theoretical

minimum of nucleation field Hn can be calculated by following formula.

$$Hn=(3/2)\gamma/(M_s \cdot R)$$

Where γ is the energy density of domain walls. M_s is the saturation magnetizing intensity. Because the γ/M_s of Sm_2Co_{17} is one-third of that of $SmCo_5$, it is easier to nucleate in Sm_2Co_{17}. When $R=10$ nm and $R=100$ nm, from formule (1) we get Hn= 2,385 kA / m and Hn=238.5 kA / m. This shows that seriously dropping of i^{Hc} is caused by the nucleation centers larger than 100 nm. When tempered at a comparatively low temperature, i^{Hc} increases though the Sm_2Co_{17} precipitations grows. And for complete nucleation centers of Sm_2Co_{17}, the calculated numeral value of i^{Hc} is ten times larger than the actual one on the basis of micromagnetism. So we may draw a conclusion that the Sm_2Co_{17} precipitated phases themselves are not nucleation centers. The composition and structure of many Sm_2CO_{17} phases are not even[7].

4 CONCLUSION

(1) With a 100 kV HVEM and by X-ray diffraction, the observation result shows that, eutectoid decomposition occurs when $SmCo_5$ magnet is tempered from 420 ℃ to 750 ℃. A part of $SmCo_5$ phase decomposes to Sm_2Co_7 and Sm_2Co_{17}.

(2) Tempered at 420 ℃ for 20 min, though $SmCo_5$ has decomposed, the coercivity shows no drop. So it shows that the eutectoid decomposition of $SmCo_5$ is not the root cause of i^{Hc} dropping at 750 ℃.

(3) Through studing on photoelectron energy spectrum, we know that no oxygen peak occurs to $SmCo_5$ samples tempered at 750 ℃.

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