Rare Met. (2019) 38(3):238–244
https://doi.org/10.1007/s12598-018-1076-0

RARE METALS
www.editorialmanager.com/rmet

Temperature stability of SmCo (2:17) magnets modified by Ni–Cr two-layer coating

Saira Bibi, Jing-Min Wang*, M. Faisal Rathore, Cheng-Bao Jiang

Received: 21 March 2017 / Revised: 19 April 2017 / Accepted: 21 May 2018 / Published online: 4 July 2018

Abstract The oxidation resistance behavior of SmCo (2:17)-type high-temperature magnets modified with Ni–Cr two-layer coating was studied. The study depicts the mass gain kinetics and magnetic properties of uncoated and Ni–Cr-modified magnets oxidized at high temperature (500 °C) in air for 200 h. The oxidation test results illustrate that the mass gain of uncoated magnet is 6.95 $\mathrm{mg \cdot cm^{-2}}$ which is more than that (0.08 $\mathrm{mg \cdot cm^{-2}}$) of coated magnet after 200 h. For the magnetic properties concerned, there is a great loss for uncoated magnet, while for coated magnet, magnetic properties do not change much. The study of uncoated magnet through X-ray diffraction (XRD) and electron probe microanalysis (EPMA) shows that the invasion of oxygen at high temperature leads to the loss of magnetic properties by changing the microstructure of magnet.

Keywords SmCo (2:17) magnets; Surface modification; High temperature stability; Ni–Cr coating

1 Introduction

Recently developed SmCo-based rare-earth permanent magnets possess potential service applications in electric aircraft and magnetic bearings for their maximum energy product ($(BH)_{\max}$) and large Curie temperature. Particularly, 2:17-type SmCo magnet has been receiving considerable attention due to high-temperature applications and their maximum operating temperature ($T_{\max}$), which can be enhanced at room temperature by improving the intrinsic coercivity or decreasing the temperature coefficient of coercivity [1–7]. Many efforts have been made by adjusting the composition (Sm, Cu, Fe) to achieve high performance in quinary system Sm(Co, Cu, Fe, Zr)$_z$. And previous works have confirmed that the 2:17-type SmCo magnets exhibit large $(BH)_{\max}$ at comparative high temperature [8–11]. However, above 500 °C, due to the severe surface oxidation (internal and external), the metallurgical changes occur that ultimately cause the irrecoverable loss of magnetic properties [12–15]. To overcome oxidation problem previously, different techniques were carried out which involve the addition of foreign alloying elements to the substrate [8, 9] and surface modification. But the former method deteriorates the intrinsic magnetic properties (i.e., remanence and energy product) of magnets as well.

A viable method is to isolate the magnet surface with some protective coatings, and earlier studies showed that surface modification is the best method without the loss of magnetic properties. Saunders and Nicholls [16] investigated some coating schemes to protect high-temperature oxidation. Several electrolytic and electroless metal coatings (Al, Ni, Ag and W) have been studied by Chen et al. [17, 18], which showed that coatings are beneficial for stabilizing the magnetic properties of 2:17-type SmCo magnet at elevated temperature from 300 to 550 °C. Wang et al. [19] prepared Ni coating on SmCo magnets and investigated mass gain kinetics and magnetic properties, also achieving the protection from oxidation. But the oxides formed at high temperature (NiO and WO_3) have unprotected and porous nature which allow further

S. Bibi, J.-M. Wang*, C.-B. Jiang
School of Materials Science and Engineering, Beihang University, Beijing 100191, China
e-mail: jingmin@buaa.edu.cn

M. Faisal Rathore
National Center of Physics, Quaid-e-Azam University, Islamabad 45320, Pakistan

penetration of oxygen with time. Pragnell et al. [20, 21] worked on oxidation protection of Sm_2Co_{17} alloys by surface modification (Pt, SiO_2, etc.) but without giving the scientific explication of results. Cr reacted with oxygen and formed Cr_2O_3 which is an excellent oxygen-diffusion barrier at high temperature even as high as 1000 °C. Dong et al. [22] and Zhao et al. [22] applied Cr and Cr_2O_3 films to improve oxidation resistance behavior of 2:17-type SmCo magnets by using double glow sputtering and arc ion-plated techniques, respectively, which are quite expensive methods.

In this report, Ni–Cr two-layer coating was fabricated by electroplating to enhance the oxidation resistance. It is not easy to deposit Cr directly on the substrate due to acidic bath composition which dissolves substrate. Ni coating provides a barrier between substrate and Cr coating. The results showed that due to the addition of Cr coating layer on Ni, thermally grown Cr_2O_3 scale not only prevents the ingress of oxygen at high temperature from environment while giving a decorative look but also holds firm bonding strength and restoring ability even after its damage by chipping.

2 Experimental

Alloy with nominal composition Sm $(Co_{bal}Fe_{0.1–0.2}Cu_{0.06–0.10}Zr_{0.033})_{7.6}$ was prepared by powder metallurgy method followed by heat treatment process. The compacts from ball-milled powder formed at 200 MPa after magnetic field orientation (5 T), under argon atmosphere, were sintered and solution-treated at 1200–1220 °C for 5 h, followed by isothermal aging at 800–820 °C for 24 h and then slow-cooling to 400 °C at the rate of 0.5 $°C\cdot min^{-1}$ and at 400 °C isothermal aging for 10 h. Finally, water quenching was carried out to room temperature. Samples with dimensions of (Φ10 mm × 10 mm) were surface modified using simple electrolytic process by Ni coating (Ni plating bath composition: 250–300 $g\cdot L^{-1}$ $Ni_2SO_4\cdot 6H_2O$, 80–100 $g\cdot L^{-1}$ Na_2SO_4, 8–12 $g\cdot L^{-1}$ NaCl, 35–45 $g\cdot L^{-1}$ H_3BO_3, 0.3–0.5 $g\cdot L^{-1}$ $C_4H_6O_2$, 0.01–0.05 $g\cdot L^{-1}$ $C_{12}H_{22}SO_3Na$ with pH = 5.0–5.5, current density of 0.5–1.0 $A\cdot dm^{-2}$, temperature of 44 °C and time of 30 min) and Cr coating (Cr bath composition: 200–250 $g\cdot L^{-1}$ Cr_3O, 2.0–2.5 $g\cdot L^{-1}$ H_2SO_4 with pH = 3.5–4.0, current density of 40–60 $A\cdot dm^{-2}$, temperature of 55 °C and time of 5–10 min). Electroplating of Ni (undercoat) and Cr (topcoat) coating was performed on the bare magnet samples. Prior to the application of plating, electrolytic degreasing was carried out to remove dust and rust from samples surface.

Uncoated and Ni–Cr-coated samples (Φ10 mm × 10 mm) were oxidized in air at 500 °C for 200 h. During oxidation test, samples were cooled at room temperature and tested at different time intervals at room temperature and 500 °C for the measurements of magnetic properties (NIM-500C) and mass gain. Microstructure study of coated and uncoated magnets (Φ5 mm × 2 mm) was carried out using electron probe microanalysis (EPMA, Model No JXA8100) with energy-dispersive spectrometry (EDS) and X-ray diffractometer (XRD, Rigaku D/max2500PC) techniques.

3 Results and discussion

3.1 Mass gain kinetics

Mass gain kinetics of 2:17 type SmCo magnets (Φ10 mm × 10 mm) with and without coating (Ni–Cr) after 200-h air oxidation at 500 °C recorded at specific time interval are shown in Fig. 1. From Fig. 1, oxidation behavior of magnets with and without coatings is clear. The mass gain of uncoated magnet increases with time, demonstrating that the magnets surface was oxidized seriously at this temperature, which is consistent with the results of previous studies [19, 23–27]. As illustrated in Table 1, uncoated sample shows 6.95 $mg\cdot cm^{-2}$ continuous increase of mass gain during 200-h oxidation at 500 °C. As for Ni–Cr-coated sample, mass gain after 200-h oxidation is only 0.08 $mg\cdot cm^{-2}$. And this mass gain becomes constant after 30-h oxidation for coated sample and this behavior is comparable with those of Cr (oxidation constant ~ 7.9×10^{-12} $g^2\cdot cm^{-4}\cdot s^{-1}$) and Cr_2O_3 (oxidation constant ~ 9.9×10^{-12} $g^2\cdot cm^{-4}\cdot s^{-1}$) coatings in Refs. [22, 28], thus showing the superiority of coating compared with that of uncoated magnet.

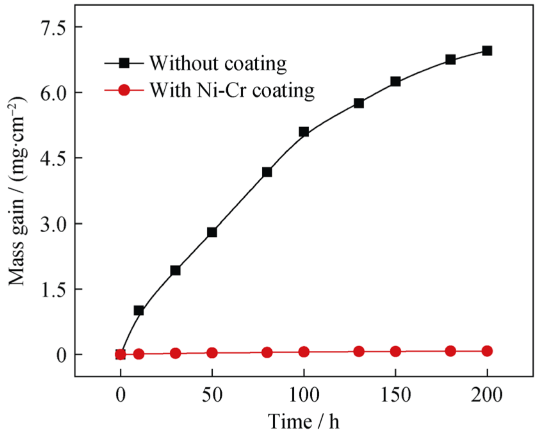

Fig. 1 Mass gain kinetics of $Sm(Co_{bal}Fe_{0.1–0.2}Cu_{0.06–0.10}Zr_{0.033})_{7.6}$ magnet oxidized at 500 °C

Table 1 Mass gain of $Sm(Co_{bal}Fe_{0.1–0.2}Cu_{0.06–0.10}Zr_{0.033})_{7.6}$ magnet oxidized at 500 °C

Time/h	Mass gain of uncoated magnet/($mg\cdot cm^{-2}$)	Mass gain of Ni–Cr-coated magnet/($mg\cdot cm^{-2}$)
0	0	0
100	5.18	0.06
200	6.95	0.08

3.2 Magnetic properties

Table 2 shows the magnetic properties of uncoated and coated magnets oxidized at 500 °C at different time intervals. At room temperature, the uncoated magnet has remanence (B_r) of 0.91 T, coercivity (H_{cb}) of 0.77 T and $(BH)_{max}$ of 145.8 $kJ\cdot m^{-3}$, while at high temperature (500 °C), the values of B_r, H_{cb} and $(BH)_{max}$ are 0.65, 0.45 T and 67.10 $kJ\cdot m^{-3}$, respectively. After 200-h oxidation at 500 °C, the magnetic properties at room temperature of uncoated magnet decreased to 0.80, 0.69 T and 104.00 $kJ\cdot m^{-3}$ with the fall of 12.0%, 10.3% and 28.6% in respective properties. But at 500 °C after 200-h oxidation, the magnetic properties are reduced to 0.51, 0.30 T and 41.00 $kJ\cdot m^{-3}$ with 21.5%, 33.3% and 38.8% drop, respectively, as shown in Fig. 2.

As for coated magnets concerned, the initial values of B_r, H_{cb} and $(BH)_{max}$ at room temperature are 0.91, 0.77 T and 145.7 $kJ\cdot m^{-3}$ and at 500 °C, these magnetic properties are 0.65, 0.45 T and 67.82 $kJ\cdot m^{-3}$, respectively. After 200-h oxidation at room temperature, the values of B_r, H_{cb} and $(BH)_{max}$ are lessen to 0.90, 0.75 T and 143.2 $kJ\cdot m^{-3}$ with 1.09%, 2.6% and 1.7% drop, respectively. While at 500 °C after 200 h oxidation, the B_r, H_{cb} and $(BH)_{max}$ are reduced to 0.64, 0.44 T and 67.00 $kJ\cdot m^{-3}$ with the decline of 1.5%, 2.1% and 1.2%, respectively.

It is obvious that the loss of magnetic properties for uncoated sample is more compared to the coated sample, as after 100-h oxidation the magnetic properties loss becomes constant, showing the self-passivation nature of Cr_2O_3 formed at high temperature. These data further confirm that surface modification (Ni–Cr coatings) improves the long-term thermal stability of SmCo magnets, suggesting that the coatings can prevent oxidization and help the stabilization of magnetic properties obviously at room and high temperature.

In previous study, a method was established to calculate the degree of thermal stability improvement, in which the magnetic loss of uncoated magnets was used as baseline of instability (100) and relative stability (1.00) [18, 29]. According to the method, the calculation results are given in Table 3. The thermal stability of Ni–Cr-modified magnets is improved by 150% at room temperature in air after 200-h oxidation, while thermal stability improvement is 298% for Ni–Cr-modified magnet at 500 °C. From above data it can be easily concluded that surface modification method is better for improving the thermal stability. In a word, the SmCo alloys can be protected by Ni–Cr coatings from oxidation at 500 °C, which can enhance the thermal stability and magnetic properties.

3.3 Oxidation mechanism analysis

EPMA examination was carried out for samples without coating and Ni–Cr coating after 200-h oxidation at 500 °C. The alloy without coating shows significant amount of oxidation, as seen from Fig. 3a. There are obvious two oxidation layers on the alloy surface internal oxidation layer and external oxidation layer. And cross-sectional microstructure image of Ni–Cr-coated sample is given in Fig. 3b; there is diffusion zone with only 1–2 μm in thickness between Ni coating and magnet substrate which is thin and does not affect the magnetic properties of

Table 2 Magnetic properties of coated and uncoated $Sm(Co_{bal}Fe_{0.1–0.2}Cu_{0.06–0.10}Zr_{0.033})_{7.6}$ magnet oxidized at 500 °C at different time intervals

Time/h	Room temperature						High temperature (500 °C)					
	Uncoated magnets			Ni–Cr-coated magnets			Uncoated magnets			Ni–Cr-coated magnets		
	B_r/T	H_{cb}/T	$(BH)_{max}$/($kJ\cdot m^{-3}$)	B_r/T	H_{cb}/T	$(BH)_{max}$/($kJ\cdot m^{-3}$)	B_r/T	H_{cb}/T	$(BH)_{max}$/($kJ\cdot m^{-3}$)	B_r/T	H_{cb}/T	$(BH)_{max}$/($kJ\cdot m^{-3}$)
0	0.91	0.77	145.8	0.91	0.77	145.7	0.65	0.45	67.10	0.65	0.45	67.82
After plating	–	–	–	0.91	0.76	145.1	–	–	–	0.65	0.45	67.80
50	0.86	0.73	120.7	0.91	0.76	144.6	0.55	0.40	46.67	0.64	0.44	67.51
100	0.82	0.71	110.5	0.90	0.75	143.2	0.53	0.35	43.21	0.63	0.43	67.43
150	0.81	0.70	105.7	0.90	0.75	143.2	0.52	0.32	42.00	0.62	0.44	67.40
200	0.80	0.69	104.0	0.90	0.75	143.2	0.51	0.30	41.00	0.62	0.44	67.00

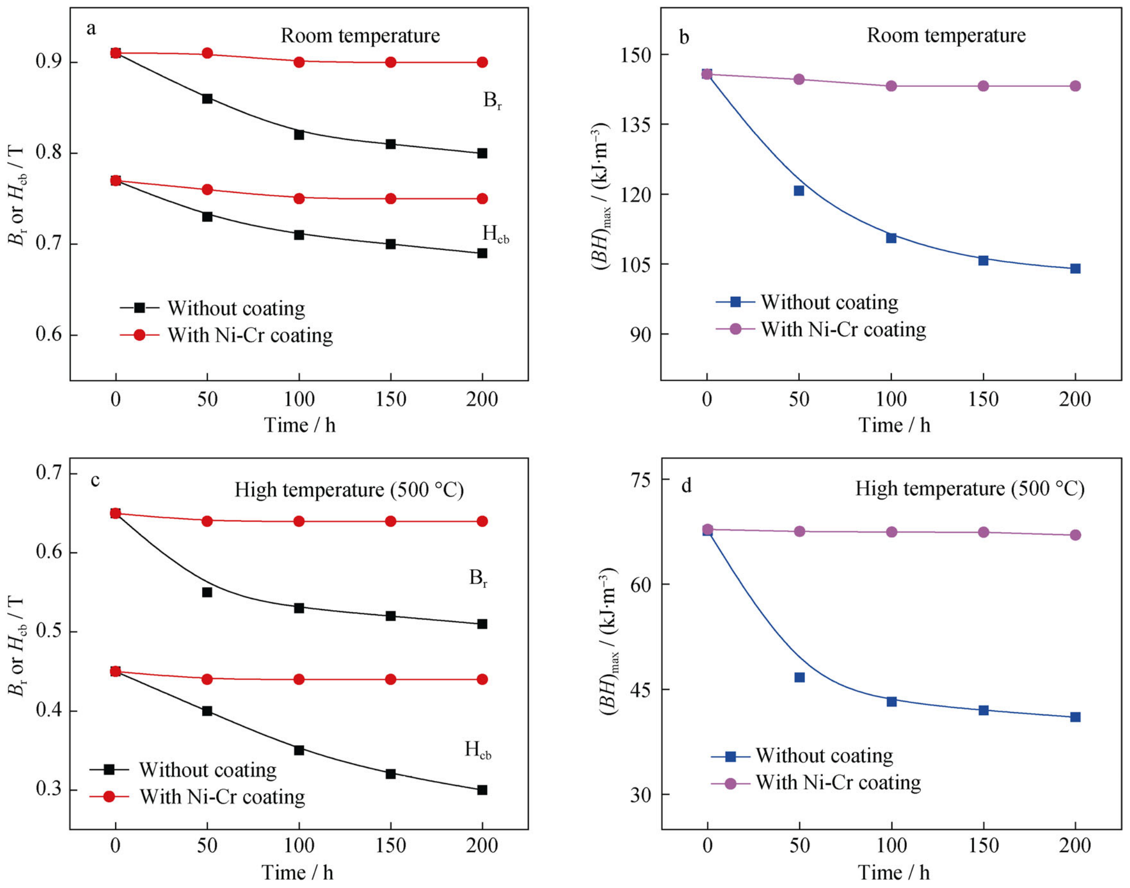

Fig. 2 Magnetic properties of $Sm(Co_{bal}Fe_{0.1-0.2}Cu_{0.06-0.10}Zr_{0.033})_{7.6}$ magnets after 200-h oxidation at 500 °C: **a** B_r and H_{cb} at room temperature, **b** $(BH)_{max}$ at room temperature, **c** B_r and H_{cb} at 500 °C, and **d** $(BH)_{max}$ at 500 °C

Table 3 High temperature stability improvement at 500 °C and its calculation method

Test condition	Condition	Magnetic loss/%	Instability scale	Stability	Relative stability	Improved stability/%
At RT in air	Uncoated	28.6	100	0.0100	1.00	0
	Ni–Cr coated	1.9	6.64	0.1506	15.05	150
At 500 °C in air	Uncoated	38.8	100	0.0100	1.00	0
	Ni–Cr coated	1.3	3.35	0.2985	29.85	298

substrate. This diffusion zone has less concentration of Ni compared to Ni coating and notorious reduction of Fe and Co concentration is observed.

XRD patterns of SmCo (2:17) uncoated sample before and after 200-h oxidation at 500 °C are given in Fig. 4. The original alloy apparently consists of 1:5 and 2:17 phases, while oxidation sample mainly contains $CoFe_2O$, CuO and Co_3O_4. There is no obvious peak of SmCo (2:17), indicating that the uncoated SmCo magnet has been oxidized remarkably at high temperature. Combined with above EPMA image, internal oxidation layer should consist of Sm_2O_3 (the most stable metal oxide) and FeCo since Sm has lower vapor pressure. Therefore, it oxidizes internally to form Sm-rich oxide in a (Co, Fe) matrix at high temperature. Internal oxidation layer increases with time into the bulk alloy at high temperature since oxygen penetrates the external oxides layer. Thus, external oxidation layer is adherent and unprotected which contains oxides ($CoFe_2O$, CuO and Co_3O_4) [12, 13].

XRD patterns of Ni–Cr-coated sample before and after 200-h oxidation at 500 °C are given in Fig. 5. At room temperature, there are sharp peaks, indicating the presence of Ni and chrome coatings. But at 500 °C after 200-h oxidation, there are a lot of peaks, indicating that at this high temperature, Cr_2O_3 is formed on the surface of coated

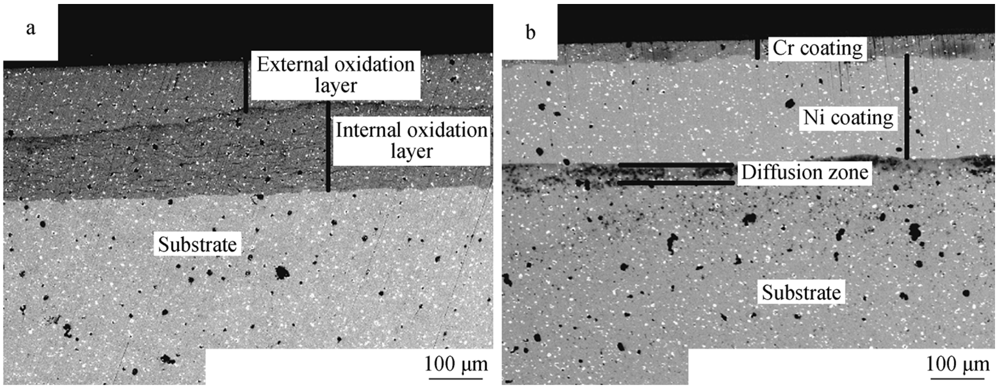

Fig. 3 Typical cross-sectional EPMA images of $Sm(Co_{bal}Fe_{0.1-0.2}Cu_{0.06-0.10}Zr_{0.033})_{7.6}$ after 200-h oxidation at 500 °C: **a** without coating and **b** with Ni–Cr coating

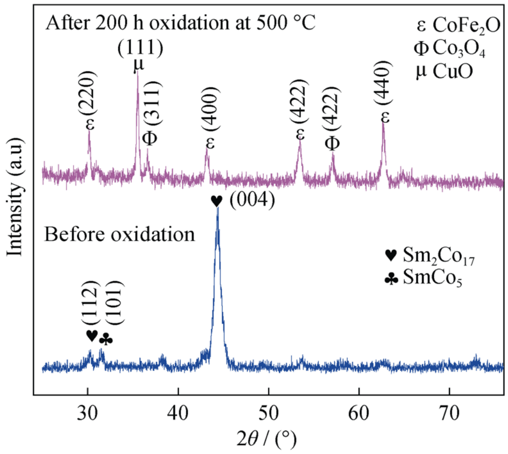

Fig. 4 XRD patterns of $Sm(Co_{bal}Fe_{0.1-0.2}Cu_{0.06-0.10}Zr_{0.033})_{7.6}$ magnet before oxidation and after 200-h oxidation at 500 °C

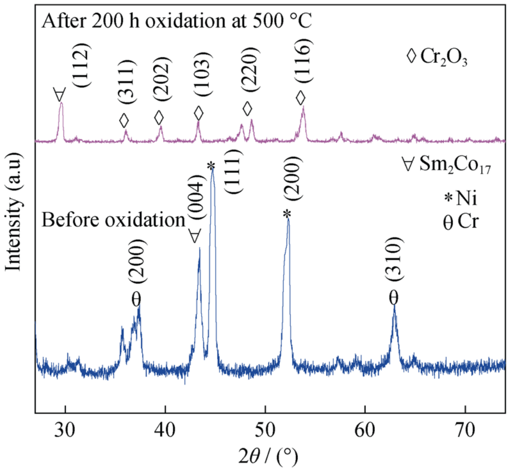

Fig. 5 XRD patterns of Ni–Cr-coated $Sm(Co_{bal}Fe_{0.1-0.2}Cu_{0.06-0.10}Zr_{0.033})_{7.6}$ magnet before oxidation and after 200-h oxidation at 500 °C

sample which acts as an obstacle to further oxidation of magnet.

4 Discussion

Mass change of uncoated magnet after 200-h oxidation at high temperature (500 °C) is almost 7 times more than that of coated magnet, as shown in Fig. 1. At this high temperature, almost 1 wt% oxygen from atmosphere penetrates substrate and 6 wt% Sm atoms are consumed. As a result, Sm_2O_3 and transition metal oxide FeCo are formed which are constitutions of internal oxidation layer. Sm_2O_3 is a nonmagnetic phase and FeCO is a soft magnetic phase, and these two phases mainly contribute to the loss of magnetic properties. External oxidation layer is composed of $CoFe_2O_4$, Co_3O_4 and CuO, which indicates that Sm atoms evaporate from substrate surface [1, 30]. EDS line profile through two coatings and magnet substrate after 200-h oxidation at 500 °C is given in Fig. 6. Four different zones are observed, including Cr coating, Ni coating, diffusion zone and magnet substrate. The outermost zone is Cr coating with ~ 5 μm in thickness, then Ni coating zone with ~ 11 μm in thickness, and then there is diffusion zone with ~ 2 μm in thickness which is also clear in Fig. 3b. This interphase diffusion zone between Ni coating and main substrate has a shape reduction of Ni concentration compared to the Ni coating area and neglectable amount of Fe and Co concentration.

Both kinds of coatings have a strong bonding with each other and with substrate as well with dense structure. For Ni coating, no cracking and spallation are observed but Cr_2O_3 is restored again at high temperature because of its strong self-passivation nature [22, 28, 31]. In case of chrome coating, oxide layer formed is dense and passive that prevents the diffusion of further oxygen into the underlying Ni coating, thus avoiding the formation of NiO.

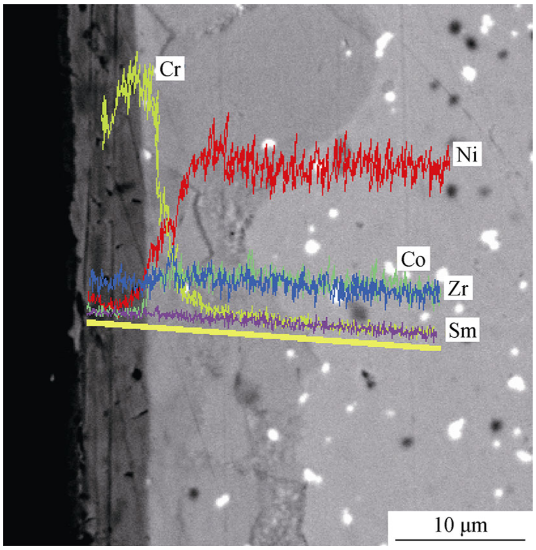

Fig. 6 EDS line profile through Cr, Ni coatings and $Sm(Co_{bal}Fe_{0.1–0.2}Cu_{0.06–0.10}Zr_{0.033})_{7.6}$ magnet after 200-h oxidation at 500 °C

The presence of Cr coating can isolate the oxygen in air at 500 °C and hinder the volatilization of Sm atoms and formation of Sm_2O_3 and FeCo. Barrier effect of chrome coating on Ni along with its passivation property provides an improvement to thermal stability of Sm_2Co_{17}-type high-temperature permanent magnets at 500 °C over a long period of time. The innermost zone is the substrate which has the same constitutions as the main magnet with homogeneous distribution of alloying elements throughout the structure.

5 Conclusion

The mass gain of uncoated sample is 7 times more than that of the Ni–Cr sample which is only 0.08 $mg \cdot cm^{-2}$, showing that Ni–Cr coating can enhance the oxidation resistance of SmCo-based high-temperature magnets. At 500 °C after 200-h oxidation when measured at room temperature for uncoated magnet, there is 28.6% lost in $(BH)_{max}$ value and 38.8% drop at high-temperature measurement, while for Ni–Cr-coated magnet they are 1.7% and 1.2%, respectively. In chrome coating at high temperature, thermally formed Cr_2O_3 has restoring property even after spallation and has passive nature, thus protecting oxygen penetration into Ni coating, which further hinders evaporation of Sm atoms.

Acknowledgements This work was financially supported by the National Science Foundation of China (No. 51471016) and the Natural Science Foundation of Beijing (No. 2151002).

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