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Erratum: “Direct chemical synthesis of high coercivity SmCo nanoblades” [Appl. Phys. Lett. 93, 032505 (2008)]

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In the Letter of Ref. 1, the authors presented results of studies detailing the structural, chemical, and magnetic properties of chemically processed magnetic nanoparticles then believed and proposed to be metallic, ferromagnetic SmCo.

Since the time of that publication, the authors, in collaboration with other researchers, have endeavored to probe further into the properties of those materials to elucidate the relationships between their magnetic properties and processing parameters and to explore the feasibility of property optimization and large scale production.

This erratum presents recent results that importantly clarify the nature of the nanoparticles produced and reported in Ref. 1. Our principle finding is that the primary phase(s) of these particles are not intermetallic Sm_xCo_y alloys or compounds, as first believed, but instead are intermetallic cobalt carbides Co_xC .

Figure 1 presents a representative laboratory $\theta-2\theta$ x-ray diffraction (XRD) scan (Cu $K\alpha$ radiation, $\lambda=0.15418$ nm) obtained from powder processed using the modified polyol method detailed in Ref. 1. It is noted that the XRD data constitutes a pattern that is especially broad and complex with a high background signal, as expected from nanoscaled intermetallic compounds synthesized by wet chemistry techniques. The top panel of Fig. 1(a) displays the raw data collected at room temperature from powder that was chemically processed, rinsed, and dried. Beneath this panel are four data sets, i.e., Figs. 1(b)–1(e), obtained from JCPDS powder diffraction reference files (in parentheses) in which the intensity and position of each Bragg peak is represented by a vertical line. The four files include those for Co_2C (65-1457), Co_3C (26-0450), Sm_1Co_5 (65-3473), and $\text{Sm}_2\text{Co}_{17}$ (26-0484) phases.

In Ref. 1, the authors attempted to match the Bragg peaks of the experimental diffraction data with those corresponding to combinations of phases that were judged likely to result from reaction of the precursor solutions. Candidate phases considered in the initial analyses were SmCo metal alloys, Sm and Co hydroxides, Sm and Co oxides, Sm and Co metals, and unreacted precursor phases. At that time the authors did not consider the existence of carbide phases in the synthesized powders as no experimental data suggested their presence and no previous published reports had indicated such products.

Based upon the methodology of Ref. 1, the relatively good agreement between the position and amplitude of the most prominent Bragg peaks in the diffraction range of $2\theta=40^\circ-50^\circ$, together with chemical analysis and the room temperature permanent magnetic properties, the authors concluded that the synthesized nanoparticles existed principally as SmCo intermetallic phases. The detection of unidentified diffraction features in the powder spectrum led the authors to propose in Ref. 1 that secondary phases of significant volume fraction were present. Due to the lack of information, the nature of these phases was not determined nor speculated upon at the time.

Energy dispersive x-ray spectroscopy (EDXS) measurements carried out within a scanning electron microscope reported in Ref. 1 found Sm and Co in ratios reflecting the nominal ratio of precursor chemicals and further supported the presence of SmCo phases. Opportunities to detect carbide phases were complicated by the use of carbon tape to reduce

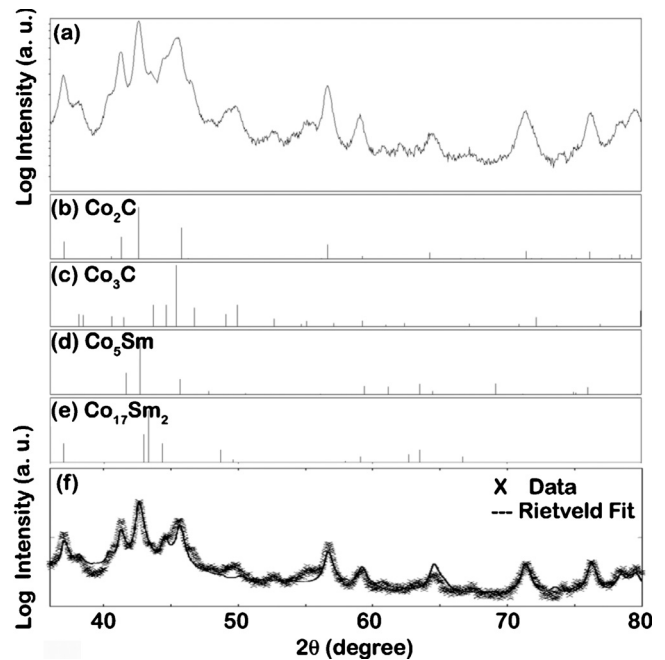


FIG. 1. Composite figure of XRD data. (a) $\theta-2\theta$ XRD data collected at room temperature using Cu $K\alpha$ radiation. JCPDS powder diffraction reference files in which the intensity and position of each Bragg peak is represented by a vertical line: (b) Co_2C (65-1457), (c) Co_3C (26-0450), (d) Sm_1Co_5 (65-3473), and (e) $\text{Sm}_2\text{Co}_{17}$ (26-0484) phases. (f) Best fit Rietveld refinement data to raw $\theta-2\theta$ XRD data.

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surface charging during the EDXS measurements. Recently, we have employed multiple rinsing steps followed by the remeasurement of the powders to show a reduction in Sm content suggesting that the Sm existed principally as a soluble phase, likely a salt.

Figure 1(f) is an XRD Rietveld refinement best-fit comparison to the raw XRD data that verifies the presence of carbide phases and allows for the accurate determination of lattice parameters and volume fractions. While the modified polyol reactions of Ref. 1 do not result in predominantly intermetallic Sm_xCo_y , they do serendipitously result in the synthesis of cobalt carbide nanoparticles shown in Ref. 2 to have room temperature coercivities greater than 4 KOe and $(\text{BH})_{\text{max}}$ values greater than 20 KJ/m^3 for not yet optimized processing, chemistry and structure.

The presence of residual phases, as indicated by diffracted amplitudes not reproduced in the Rietveld model [see Fig. 1(f)], and their contribution to the particles' magnetic properties, has not yet been made clear. In particular, an amorphous or nanoscale SmCo phase that is below the detection limit of laboratory XRD (≤ 5 wt %) may exist and contribute to the hard magnetic properties of these powders.

Since the time of the publication of Ref. 1, the authors have performed in-depth studies including extensive processing, and chemical, structural, and magnetic measurements that have led them to conclude the powders of Ref. 1 consist primarily of cobalt carbides particles. Further refinement of the processing methodologies has shown that cobalt carbide particles, consisting of Co_2C and Co_3C , can be reproducibly obtained using the modified polyol process.²

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