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Microstructure of Ni(Cr)-TiC-Cr_3C_2-Cr_7C_3 composite powder

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Abstract: The Ni(Cr)-TiC-Cr_3C_2-Cr_7C_3 composite powder was prepared by SHS (Self-propagating High-temperature Synthesis) process using elemental powders as starting materials. As-prepared powder was ground by an attritorial mill up to 10 hours. Micrograph and diffraction patterns were obtained using TEM. The X-ray diffraction data were analyzed using the Rietveld method and Toraya procedure. The Rietveld method was applied in the determination of phase abundance using Hill and Howard approach. The parameters of diffraction line profiles were analyzed by PRO-FIT Toraya procedure. The crystallite sizes and lattice distortions were analyzed using Williamson-Hall method. It was stated that the crystallite size of Ni(Cr) phase diminishes to nanoscale after 5 hours milling whereas the crystallite size of the other phases is still above 100 nm for applied milling time.

Keywords: Composite powder, Intermetallics, Titanium and chromium carbides, XRD, TEM

1. INTRODUCTION

SHS (Self-propagating High-temperature Synthesis) process is characterized by low-processing temperatures, easy compact of powders as starting materials, flexibility in composition and relatively short process time [1]. Milling process is a technique of widespread application. In the present work this process was applied for diminishing the size of crystallites of composite powder. The components of studied material ensure its excellent high temperature corrosion resistance.

In the present work the X-ray data were analyzed using Rietveld structure refinement method [2-7] and Toraya procedure [8]. Rietveld method was applied mainly for the determination of phase abundance. Morphology of studied materials was analyzed by TEM method.

2. MATERIAL AND RESEARCH METHODOLOGY

SHS process was used for preparation of studied composite powder. As-prepared powder was milled in attritorial mill (250 rpm, stainless steel balls of 6 mm diameter, 10:1 ball-to-powder weight ratio, 2 dm^3 chamber capacity) for 2, 5 and 10 hours.

X-ray diffraction patterns were collected using X-Pert Philips diffractometer equipped with curved graphite monochromator on diffracted beam and with the following slits (in the sequence from Cu tube to counter); Soller (2^o), divergence (1/2°), antiscatter (1/2°), Soller (2^o) and receiving (0.15 mm).

The profile parameters of individual diffraction lines were determined using Toraya PRO-FIT procedure [8] which applies Pearson VII function for the description of line profiles.

The Rietveld analysis was performed applying DBWS-9807 program that is an update version of the DBWS programs for Rietveld refinement with PC and mainframe computers. The pseudo-Voigt function [4] was used in the describing of diffraction line profiles at Rietveld refinement. The quantitative phase analysis was performed using the relation proposed by Hill and Howard [9].

The crystallite sizes and lattice distortions for Ni(Cr) phase were estimated using Williamson-Hall method [10]. The NIST SRM660a (LaB_6 powder) was used as line profile standard for instrumental broadening determination.

For TEM analysis powders were suspended in ethanol and scattered for few minutes in an ultrasonic bath before being collected on a carbon grid. TEM studies were performed using JEOL 3010 microscope operated at 300 kV..

3. RESULTS AND DISCUSSION

Detailed analysis of X-ray diffraction data of as-prepared powder reveals the presence of Ni(Cr), TiC, Cr_3C_2 and Cr_7C_3 phases (Fig. 1). The contents of these phases determined by Hill and Howard procedure are 22.0, 38.3, 24.5 and 15.2 wt.%, respectively.

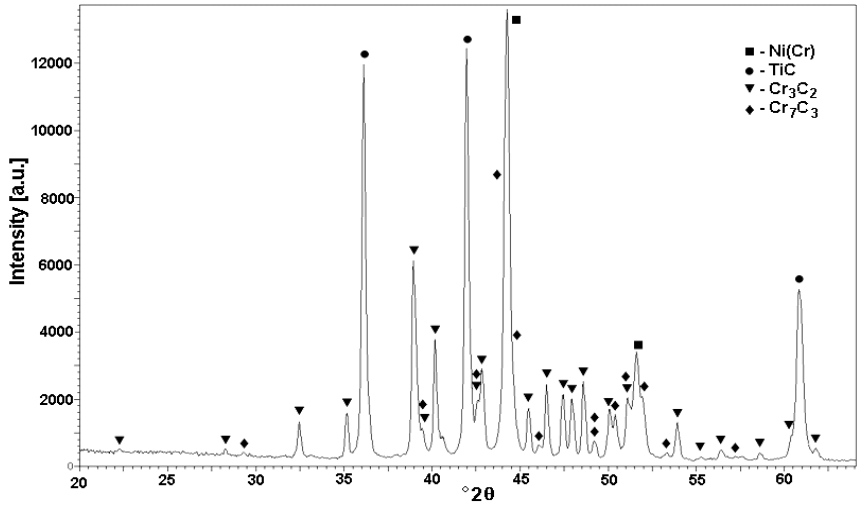

Figure 1. XRD pattern of as-prepared sample

The broadening of diffraction lines for samples ground at prolonged times are observed mainly for Ni(Cr) phase as can be clearly seen in Fig. 2 which presents the changes of FWHM parameters of selected diffraction lines for Ni(Cr) and TiC phases. For reference the FWHM parameters of LaB_6 standard are also presented.

The decrease of crystallite size to nanoscale (87 nm) is observed only for Ni(Cr) phase in the sample milled at least for 5 hours. The crystallite size diminishes to 45 nm with the increase of milling time to 10 hours. TEM studies (Fig. 3) verify the X-ray diffraction analysis.

The broadening of diffraction lines of TiC phase is much lower than observed for Ni(Cr) phase (Fig. 2). Thus the size of TiC crystallites does not change meaningful even after 10 hours milling (Fig. 3). The broadening of diffraction lines of chromium carbides (not presented in Fig. 2) is negligible. As a consequence the decrease of crystallite size of all involved carbides is negligible.

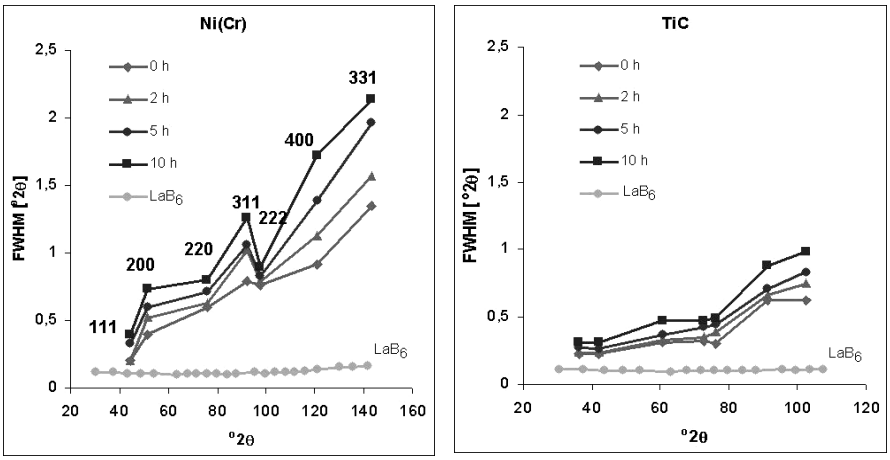

Figure 2. Changes of FWHM parameters of diffraction lines for Ni(Cr) and TiC phases as a result of milling process (indices of diffraction lines for Ni(Cr) phase are given)

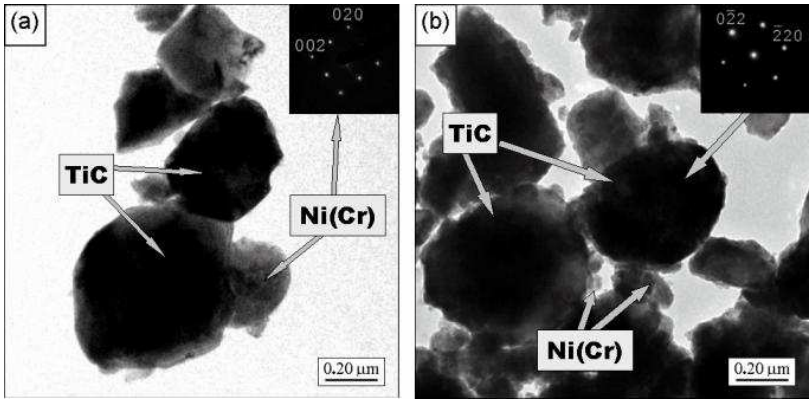

Figure 3. Bright-field TEM images of as-prepared sample (1a) and milled for 10 hours (1b) and corresponding electron diffraction patterns taken from Ni(Cr) and TiC particles along [111] and [100] zone axis, respectively

4. CONCLUSIONS

- Ten hours milling of studied composite powder results in the decrease of Ni(Cr) crystallite size up to 45 nm, leaving the crystallite size of TiC, Cr_3C_2 and Cr_7C_3 phases above 100 nm.
- From presented results it can be concluded that hard particles of TiC, Cr_3C_2 and Cr_7C_3 carbides may play an additional role of grinding media.

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