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Dispersion analysis of NiAl-TiC-Al_2O_3 composite powder

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The NiAl–TiC–Al_2O_3 ceramic, composite powder was prepared by SHS (Self-propagating High-temperature Synthesis) method. As-prepared powder was ground by high-energy attritory mill for up to 40 hours. The Rietveld method based on X-ray powder diffraction data was applied for the verification of qualitative and quantitative phase compositions of as-prepared sample. The values of lattice parameters for all the phases present in as-prepared composite powder were also established and found to be in good agreement with these found in ICDD files. From the detailed analysis of the integral breadth of diffraction lines for all phases it was stated that during high-energy milling the significant decrease of crystallite size is observed only for NiAl phase. The crystallite size of this phase diminishes from 40 nm to 31 nm with the increase of milling time from 10 to 40 hours.

1. INTRODUCTION

Rietveld structure refinement method [1-3] appeared to be very helpful not only in the microstructure characterisation (crystallite sizes, lattice distortions, dislocation densities, stacking faults and twins analysis [4, 5]) of various materials but also in the verification of the qualitative and quantitative phase compositions [6]. When detail information on the structure of concerned phases is available then quantitative estimation of phase abundance in multiphase material is possible [7].

The purpose of the present work was verification by Rietveld method based on X-ray powder diffraction data of qualitative and quantitative phase compositions of ceramic powder prepared by SHS (Self-propagating High-temperature Synthesis) [8-10] method. Moreover, the changes of crystallite sizes of phases were analysed. Studied composite, ceramic powder was intended for thermal spraying of coatings.

2. MATERIALS AND EXPERIMENT

The chemical compositions of initial mixtures were adequately selected to obtain by SHS method the desired composition of composite powder with NiAl–TiC–Al_2O_3 phases. As-prepared powder was ground in high-energy attritory mill. Samples are designated with the corresponding milling time (0h for the starting as-prepared powder).

X-ray diffraction pattern were collected using X-Pert Philips diffractometer equipped with 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 R_{wp} (weighted-pattern factor) and S (goodness of fit) parameters were used as numerical criteria of the quality of the fit of calculated to experimental diffraction data. The relative weight fractions of crystallite phase present in the studied composite materials were calculated using relation proposed by Hill and Howard [7]. The crystallite size of NiAl phase was estimated using Hall method [11]. The NIST SRM660a (LaB_6 powder) was used as line profile standard for instrumental broadening.

3. RESULTS AND DISCUSSION

Detailed analysis of the X-ray diffraction pattern of as-prepared powder shows the presence of the following phases: NiAl–TiC–Al_2O_3 (inset in Fig. 1).

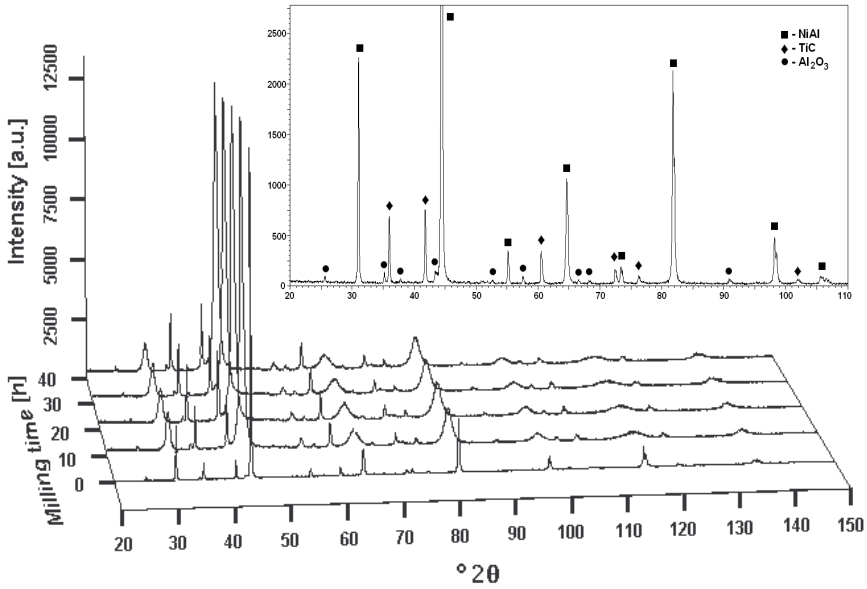

Fig. 1. Evolution of XRD patterns as a result of milling process for different time

Rietveld output of X-ray pattern of as-prepared powder for above three phases (Fig. 2) verifies this conclusion. Good agreement between calculated pattern and experimental one was obtained (the values of R_{wp} and S parameters are equal to 14.8% and 1.31, respectively) and there is no additional diffraction lines on residual curve (Fig. 2). Moreover, the values of lattice parameters that determined by Rietveld procedure (Tab. 1) appeared to be in good relation to those found in ICDD files. Using Hill and Howard procedure [7], the relative weight fractions of crystallite phase present in the studied material were calculated (Tab. 1).
Assuming that diffraction line broadening originate mainly from the decrease of crystallite size the changes of the integral breadth of selected diffraction lines (Fig. 3) clearly show that during high-energy milling the significant decrease of crystallite size is observed only for NiAl phase. The crystallite size of this phase determined using Hall procedure [11] diminishes from 40 nm to 31 nm with the increase of milling time from 10 to 40 hours.

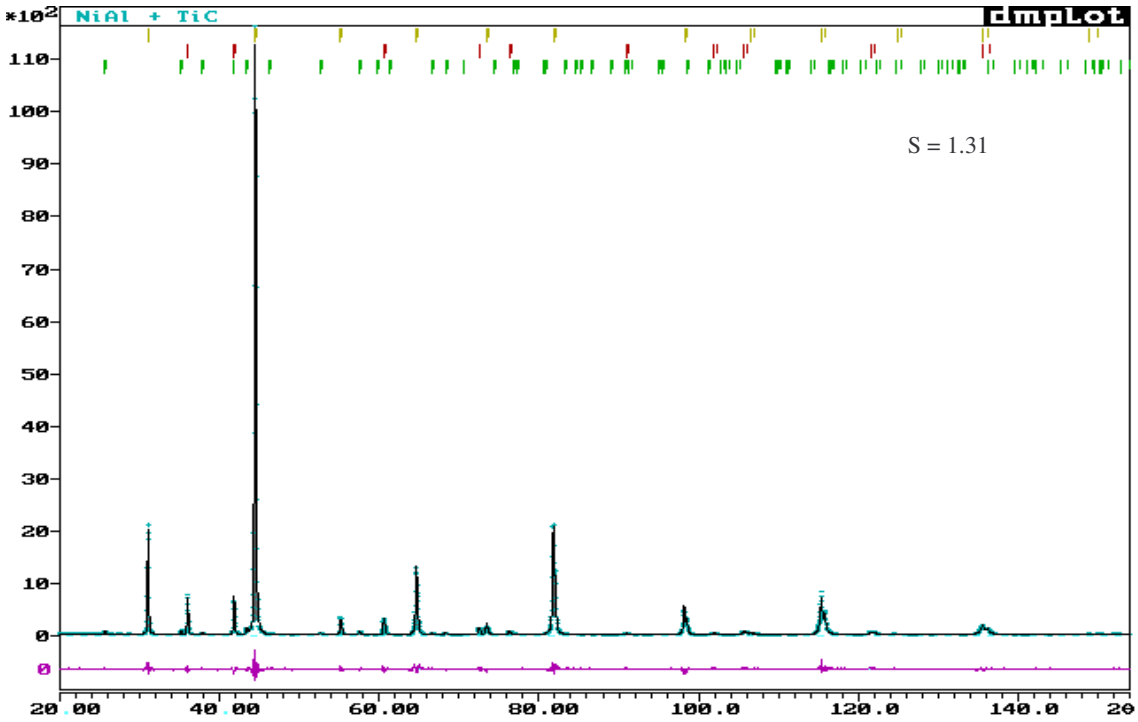

Fig. 2. Rietveld output of X-ray diffraction pattern of as-prepared sample

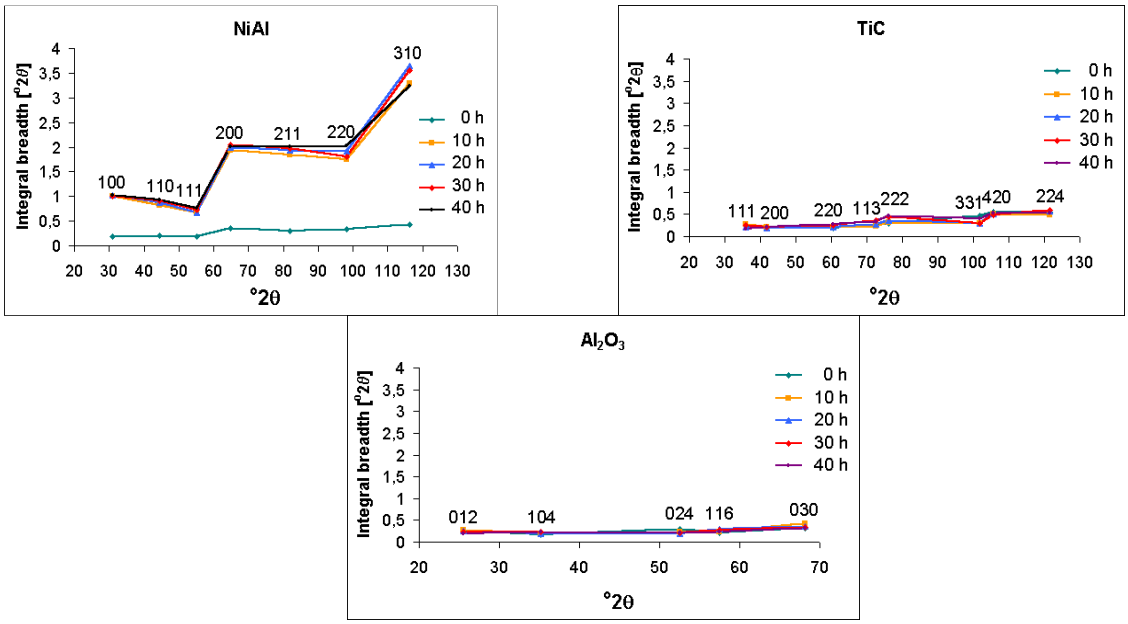

Fig. 3. Changes of the integral breadth of diffraction lines for NiAl, TiC and Al_2O_3 phases, respectively as a result of milling process (indices of diffraction lines are given)

Table 1
Comparison of lattice parameters estimated by Rietveld method and these from ICDD files and the quantitative phase composition of as-prepared powder

Component	Lattice parameters [Å]		Contents [wt.%]
	Rietveld	ICDD file	
NiAl	$a_0 = b_0 = c_0 = 2.8822$	$a_0 = b_0 = c_0 = 2.887$	86.4
TiC	$a_0 = b_0 = c_0 = 4.3218$	$a_0 = b_0 = c_0 = 4.3285$	9.6
Al_2O_3	$a_0 = b_0 = 4.7541$ $c_0 = 12.994$	$a_0 = b_0 = 4.758$ $c_0 = 12.991$	4.0

4. CONCLUSIONS

- The presence of NiAl, TiC and Al_2O_3 phases in composite, ceramic powder obtained by SHS method was stated.
- Rietveld procedure appeared to be very useful in the verification of qualitative phase composition and in the determination of quantitative abundance of phases in complex, composite powders.
- During milling of as-prepared powder the significant decrease of crystallite size was stated only for NiAl phase.

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