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## Rietveld analysis of intermetallic phases from Ni-Al system

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The Rietveld method was applied for the verification of the phase compositions of materials from Ni-Al system. Three powder materials were prepared by SHS method. It was stated that two materials contained only one phase NiAl and Ni<sub>3</sub>Al respectively, whereas the third material besides Ni<sub>2</sub>Al<sub>3</sub> phase contained also small amount of NiAl one (1.0 wt. %). The lattice parameters of all phases estimated by Rietveld method were in good agreement with these from ICDD files.

### 1. INTRODUCTION

The knowledge of qualitative and quantitative phase compositions of materials is of prime importance in materials science. Technology of materials may be very different. The Rietveld [1,2] method based on powder diffraction data appeared to be very useful in the verification of qualitative and quantitative phase compositions of materials prepared in different manner. The Rietveld method is also potentially a powerful tool for crystal refinement based on powder diffraction data. It is especially important in materials science since composition of many materials very often is not stoichiometric. In the Rietveld method structure parameters of phases are calculated fitting the entire powder diffraction pattern by the least-squares method.

The aim of the present work was the verification by Rietveld method of qualitative and quantitative phase compositions of materials prepared by SHS method [3,4,5]. This verification was performed for materials from Ni-Al system.

### 2. RESEARCH METHODOLOGY

The qualitative phase composition of materials by Rietveld method can be estimated by comparison of calculated diffraction pattern with experimental one. The appearance of additional diffraction lines on experimental pattern is an indication that material contains more than one phase. The quantitative phase composition can be also determined by Rietveld method. According to Hill and Howard [6], the quantitative phase analysis is possible directly from scale factors of the respective calculated intensities.

Relative weight functions of crystalline phases in a multiphase material can be calculated from relation:

$$W_p = \frac{S_p (Z \cdot M \cdot V)}{\sum_{i=1}^n S_i (Z \cdot M \cdot V)_i} \cdot 100\% \quad (1)$$

where:

$W_p$  – relative weight fraction of phase p in the mixture of  $n$  phases (wt. %),

$S$  – Rietveld scale factor,

$Z$  – number of formula units per unit cell,

$M$  – mass of the formula unit (in atomic mass units),

$V$  – unit cell volume (in Å<sup>3</sup>) The application of Rietveld method in quantitative phase analysis is very convenient in comparison with traditional methods, which apply only small set of integrated intensities. First of all in the Rietveld method the scale factors are estimated from all diffraction pattern; moreover the other parameters in equation (1) are available as simple parameters.

The most often used numerical criteria of the fit of calculated to experimental data are:

$$R_{wp} = \left\{ \frac{\sum w_i (y_i(obs) - y_i(calc))^2}{\sum w_i (y_i(obs))^2} \right\}^{\frac{1}{2}} 100\% \text{ - weighted-pattern } R \text{ factor} \quad (2)$$

where  $y_i(obs)$  and  $y_i(calc)$  are observed and calculated intensities respectively, and  $w_i$  the weight.

The goodness of fit ( $S$ ) is estimated from relation:

$$S = \frac{R_{wp}}{R_{exp}} \quad (3)$$

$$\text{where: } R_{exp} = \left[ \frac{N - P}{\sum w_i y_i(obs)^2} \right]^{\frac{1}{2}} 100\% \text{ - expected } R \text{ factor,} \quad (4)$$

$N$  – number of intensity data,

$P$  – number of refined parameters.

Detailed recordings of X-ray patterns were performed using X-Pert Philips diffractometer. CuK<sub>α</sub> radiation was applied. X-ray goniometer was equipped with graphite monochromator on diffracted beam and with Soller slits both on incident and diffracted beams. X-ray generator operated at 40kV and 30mA. Counting time was equal to sec for steps of 0.04° in  $2\theta$ .

### 3. RESULTS AND DISCUSSION

The verification that two materials contained only one phase NiAl and Ni<sub>3</sub>Al can be found in Fig. 1 and in Fig. 2 respectively. On the other hand Fig. 3 clearly show that the third material besides Ni<sub>2</sub>Al<sub>3</sub> phase contains also small amount of NiAl one. The estimated according to equation (1) the concentration of NiAl phase was equal to 1.0 wt. %.

The best fitting of calculated to experimental data was obtained for material with NiAl phase but generally all fittings are quite similar for all materials (Tab.1). The lattice

parameters of all phases estimated by Rietveld method are in good agreement with these found in ICDD files (Tab. 1).

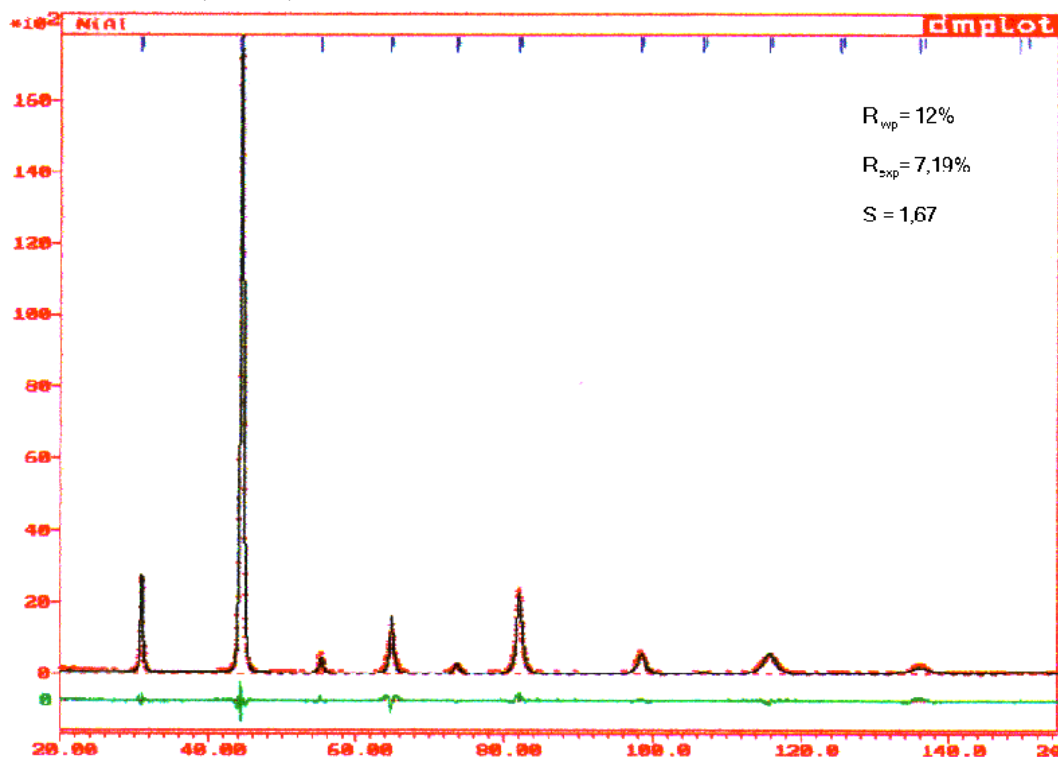


Fig. 1. Rietveld refinement plot for material with NiAl phase

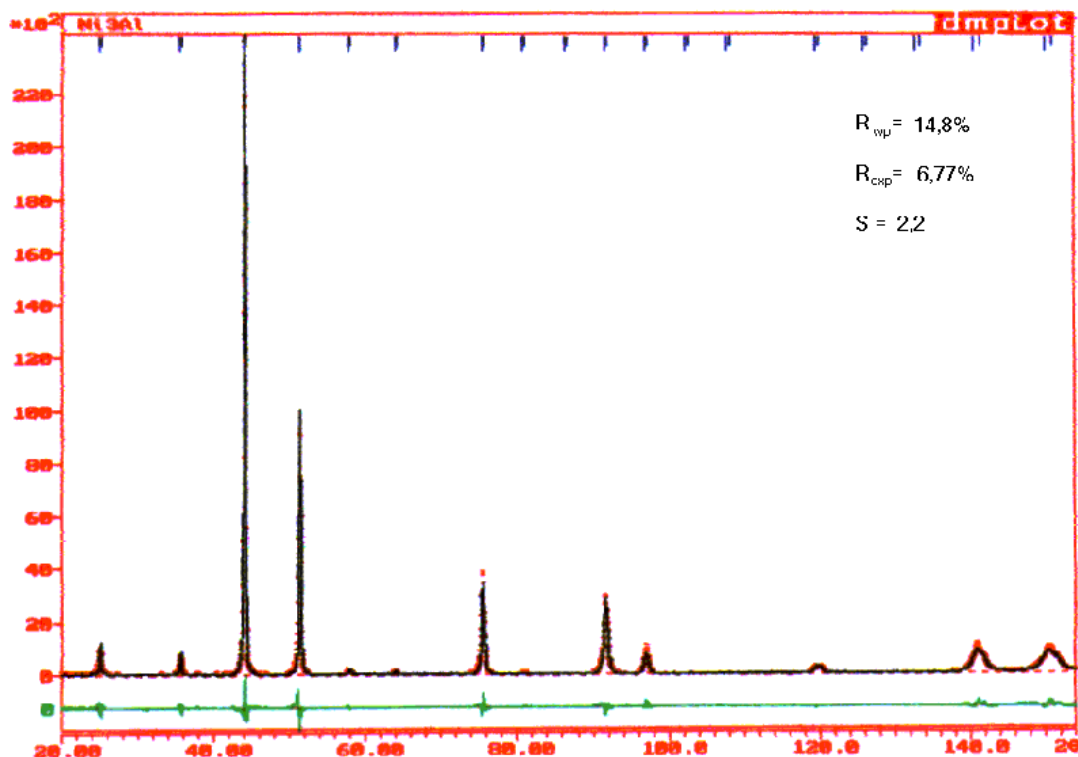


Fig. 2. Rietveld refinement plot for material with Ni<sub>3</sub>Al phase

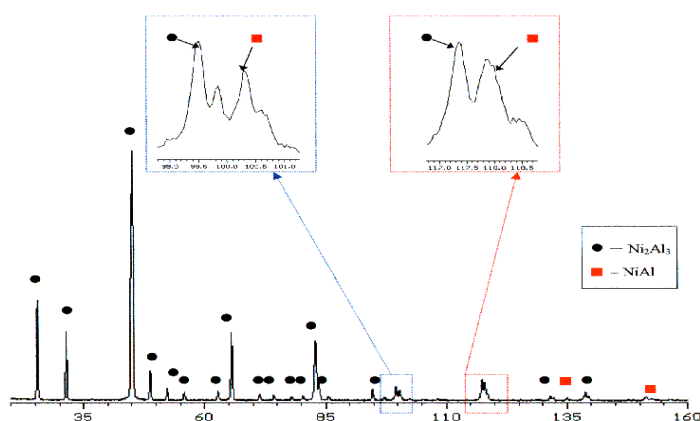


Fig. 3. X-ray diffraction pattern of two-phase material with  $\text{Ni}_2\text{Al}_3$  and  $\text{NiAl}$  phases

Table 1

Comparison of lattice parameters estimated by Rietveld method and these from in ICDD files and parameters of fitting of calculated to experimental data.

Material	Lattice parameters		$R_{wp}$	$R_{exp}$	S
	RIETVELD	ICDD			
NiAl	$a_0 = 2.8782 \text{ \AA}$	$a_0 = 2.888 \text{ \AA}$	12.0 %	7.2%	1.67
$\text{Ni}_2\text{Al}_3$ + NiAl	$a_0 = b_0 = 4.0364 \text{ \AA}$ $c_0 = 4.8974 \text{ \AA}$	$a_0 = b_0 = 4.036 \text{ \AA}$ $c_0 = 4.9 \text{ \AA}$	14.5%	7.5%	1.93
$\text{Ni}_3\text{Al}$	$a_0 = 3.5672 \text{ \AA}$	$a_0 = 3.561 \text{ \AA}$	14.8 %	6.8%	2.18

#### 4. CONCLUSIONS

Using the Rietveld method the verification of qualitative and quantitative phase compositions of powder materials from Ni-Al system obtained by SHS method was performed. It was stated that two materials contained only one phase, NiAl and  $\text{Ni}_3\text{Al}$  respectively. On the other hand the third material besides the main  $\text{Ni}_2\text{Al}_3$  phase contained small amount (1.0 wt. %) of NiAl phase. The values of lattice parameters of all phases present in studied materials were also established and were in good agreement with these from ICDD files.

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