EVALUATION OF RIETVELD METHOD APPLIED TO XRD PATTERNS FOR MICROSTRUCTURAL CHARACTERIZATION OF MAX-PHASES OF Ti-Al-C SYSTEM

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\[ \text{MAX phases}\]

MAX phases are carbides and nitrides that bridge the gap between properties of typical metals and ceramics due to their layered hexagonal crystal structures their alternating strong covalent M-X and relatively weak metallic M-A bonds [1, 2].

The aim of this project is to characterize the microstructure (preferred orientation, weight fraction and crystallites size) of Ti-Al-C MAX phases by using X-Ray diffraction technic coupled to Rietveld analysis and to conclude on the reliability (repeatability and reproducibility) of the method.

Rietveld analysis is a refining method of parameters describing the crystalline structure. It is based on the comparison and minimization of the difference of the peaks intensities of an experimental XRD pattern and a simulated theoretical diffractogram:

\[ I_{1}^{\text{calc}} = S F \sum_{j=1}^{N_{\text{phases}}} \sum_{k=1}^{N_{\text{peaks}}} f_{j} V_{j} \left( G_{j}(2 \theta_{i} - 2 \theta_{k}) P_{j} / A_{j} L_{i} + bk_{i} \right) \]

\[ S = \left( \frac{R_{\text{exp}}}{R_{\text{calc}}} \right)^{2} \]

\[ S \rightarrow 1 = \text{good refinement} \]

**Ti/Al/C MAX Phase elaboration by SPS process (BCRC)**

Simultaneous application of uniaxial pressure and pulsed electric current directly on the sample

**XRD analysis**

1. Sample preparation
   - Sintered powders are pressed in the holder
2. XRD acquisition (diffractometer)
3. Phases identification (EVA)

**Rietveld refinement procedure (MAUD software)**

**Applied sintering conditions**

<table>
<thead>
<tr>
<th>Mixture powders</th>
<th>T (°C)</th>
<th>P (MPa)</th>
<th>Holding time (min)</th>
<th>Rate (°C/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TIC: Al: Ti</td>
<td>1:1:1</td>
<td>1250</td>
<td>10</td>
<td>15</td>
</tr>
<tr>
<td>1275</td>
<td>15</td>
<td>15</td>
<td></td>
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<tr>
<td>1300</td>
<td>15</td>
<td>15</td>
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</tr>
</tbody>
</table>

**MAX phases:** 211 (Ti₃AlC₂), 312 (Ti₃AlC₂), 523 (Ti₃Al₅C₆), Impurities: TIC and Al₃Ti (**) [1]

**Weight fractions**

- SF and MD give practically the same results
- Difference of about 6.8% when (00l) P.O is not considered

**Conclusions:**

- WF and P\text{01l} factor calculations are more repeatable than crystallites size calculations
- P\text{01l} factor and WF are relatively reproducible: Influence of the powder sample preparation stage
- Crystallite size parameters are weakly reproducible