**INTRODUCTION**

The aim of this project is to characterize the microstructure (preferred orientation, weight fraction and crystallite size) of Ti-Al-C MAX phases by using X-ray diffraction technique coupled to Rietveld analysis and to conclude on the reliability (repeatability and reproducibility) of the method. Otherwise, Rietveld analysis is a refining method of parameters describing the crystalline structure. It is based on the comparison and minimization of the differences of the peaks intensities of:

a) an experimental XRD pattern
b) a simulated theoretical diffractogram

**Materials & Methods**

Sintering of powders
- **Mixture** (TiC/Al/Co: 1:1:1)
- **Sample preparation**
- **Diffractogram acquisition**
- **Phases identification**
- **Structure Refinement**

**Rietveld method procedure**

1. Phases specifications and instrument parameters
2. Background, scale factor and peaks positions
3. Cell parameters
4. Crystal structure
5. Peaks shapes
6. Preferred Orientation

**RESULTS AND DISCUSSIONS**

Repeatability (standard deviation evaluation) and reproducibility (comparison between $A_1$ and $A_2$) of the sample sintered at 1250°C

- The value of preferred orientation parameter (P) changes with the model used but the trends are the same.
- Good repeatability
- But relatively weak reproducibility

**Evolution of the preferred orientation parameter (P) of Ti$_2$AlC with temperature**

<table>
<thead>
<tr>
<th>Orientation factor</th>
<th>Weight fractions</th>
<th>Crystallite size</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A_1$</td>
<td>$A_1$</td>
<td>$A_1$</td>
</tr>
<tr>
<td>$A_2$</td>
<td>$A_2$</td>
<td>$A_2$</td>
</tr>
<tr>
<td>$A_3$</td>
<td>$A_3$</td>
<td>$A_3$</td>
</tr>
<tr>
<td>$A_4$</td>
<td>$A_4$</td>
<td>$A_4$</td>
</tr>
</tbody>
</table>

The major phase remains the most oriented and its orientation is more marked when the temperature increases.

**Weight fractions evolution with temperature**

- **MAX phases**
  - Weight fraction (March-Dollase)
  - MAX phases remain the major phase and its maximum weight fraction is obtained at 1275°C
  - The interval of weight fractions for all changes weakly from March-Dollase to Standard function

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>March-Dollase</th>
<th>Standard function</th>
</tr>
</thead>
<tbody>
<tr>
<td>1250</td>
<td>7.9</td>
<td>1.6</td>
</tr>
<tr>
<td>1275</td>
<td>/</td>
<td>/</td>
</tr>
<tr>
<td>1300</td>
<td>6</td>
<td>2.1</td>
</tr>
</tbody>
</table>

- **Other phases**
  - TiC, AlC remains the major phase and its maximum weight fraction is obtained at 1275°C
  - The interval of weight fractions for all changes weakly from March-Dollase to Standard function

**CONCLUSIONS**

Depending on the model, taking into account the orientation leads to very different values of the preferred orientation parameter (P). However, this consideration of the orientation has a relatively low impact on the quantitative analysis (weight fractions). This means that a big difference in the analysis of orientation does not lead to large differences in weight fractions from one model to another. Quantitative analysis is relatively reliable even for highly oriented samples regardless of the model used. The determination of the crystallite size is weakly repeatable even by applying the same procedure with the same model. The dispersion of the value of the crystallite size is quite important. The comparison of results can be done only if these results are obtained under identical conditions of modeling.

**REFERENCES**
