ESRF	**Experiment title: Kinetic studies on TiB_2-TiC-NiAl Composites obtained by Combustion Synthesis**	**Experiment number**: CH-1235
Beamline: ID-11	**Date of experiment**: from: 8/05/2002 to: 13/05/2002	**Date of report**: 14/01/2003
Shifts: 15	**Local contact(s)**: Gavin Vaughan	*Received at ESRF:*

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Report:

TiC-TiB2 composites are strategic industrial materials for the production of cutting tools. That is why not only the physical chemistry of the reactions but processing costs and raw materials choice are important as well. This was the reason why four paths in the synthesis of TiC-TiB_2 through Self-propagating High-temperature Synthesis (SHS) were explored using different raw materials:

$$(1+x)\ Ti + 2x\ B + C \rightarrow TiC + x\ TiB_2 \quad (I)$$

$$(1+x)\ Ti + x/2\ B_4C + (1-x/2)\ C \rightarrow TiC + x\ TiB_2 \quad (II)$$

$$(1+x)\ TiO_2 + x/2\ B_4C + (1-x/2)\ C + (2+2x)\ Mg \rightarrow TiC + x\ TiB_2 + (2+2x)\ MgO \quad (III)$$

$$(1+x)\ TiO_2 + x\ B_2O_3 + C + (2+5x)\ Mg \rightarrow TiC + x\ TiB_2 + (2+5x)\ MgO \quad (IV)$$

As this kind of reactions take place at high velocity, especial experimental conditions and setup were necessary. This was described previously (1). For these experiments some improvements of the setup were implemented with the help of ID-11 and ID22 beam line staff. Thanks to these improvements the acquisition times were reduced to 65 ms per diffraction pattern.

Experiments were carried out using a wave length of 0.2755 Å. Samples were pressed in disk of 20mm. in diameter and 2 mm. thickness. Diffraction patterns were acquired in fast sequences during the combustion, obtaining data between 1 and 14 º of 2θ. Good diffraction patterns with well defined diffraction peaks were obtained, which gave clues of the synthesis paths.

In all four reactions, differences in both kinetics and mechanisms were observed. Reaction (I) is a clear reaction starting with Ti and B, because the enthalpy of this reaction is higher than between Ti and C. Ti appears as a transition specimen due to the heat released during synthesis, that transforms to the cubic phase and melts before reaction.

Reaction (II) is a little more complex because is the reaction between the element and a stable compound such as B_4C. Depending on the x value it seems to appear that the mechanism change. For higher values of x

the mechanism is similar to reaction (I) i.e. TiB_2 synthesis is faster than TiC. Nevertheless if x is smaller than 2 TiC synthesis is the fastest.

Reaction (III) involves the use of two reagents less expensive, in order to obtain similar compositions, since the MgO obtained can be easily removed by acid solutions. In this case the reaction begins with the reduction of TiO_2, formation of both MgO, and cubic Ti. A further reaction of Ti with Boron carbide takes place to obtain TiC and TiB_2 in this order.

Reaction (IV) has a similar mechanism that reaction (III). Only kinetic changes have been observed.

A more detailed study on microstructure is being carried out in order to explain more clearly the mechanism. Nevertheless preliminary results obtained show that synthesis from oxides leads to a finer microstructure that from elements (figure 3).

These results will be the object of a few papers that will be published soon, with ESRF staff as co-authors.

(1) C. Curfs, I.G. Cano, G.B.M. Vaughn, X. Turrillas, A. Kvick, M.A. Rodríguez. "TiC-NiAl composites obtained by SHS: a time resolved XRD study". J. Europ. Ceram. Soc. 22 (2002) 1039-1044.

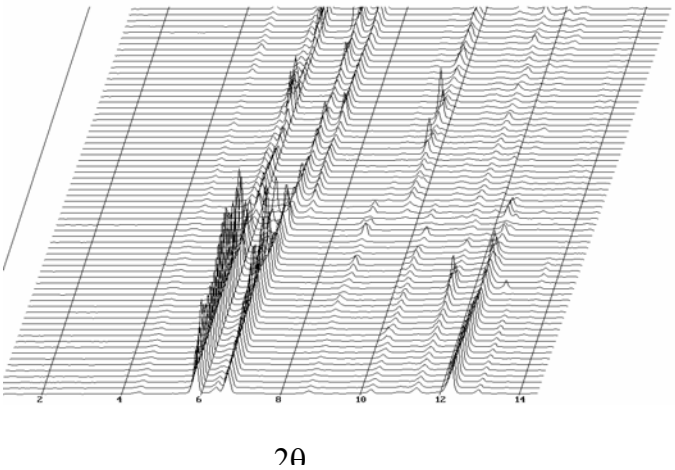

Figure 1. Diffraction patterns of reaction synthesis II.

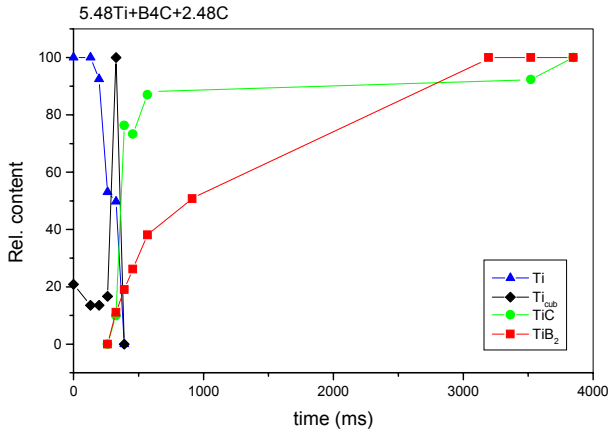

Figure 2. Intensity evolution of the most important diffraction peaks of the specimens involved in reaction II.

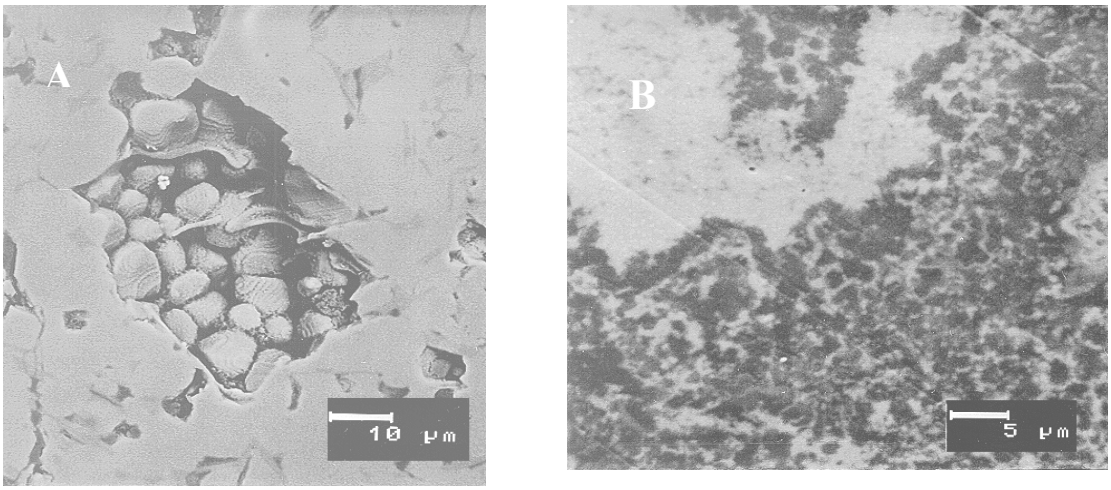

Figure 3.- Micrograph obtained by SEM of: A) materials obtained from reaction I, B) materials obtained from reaction IV.