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In-situ synchrotron X-ray diffraction study of TiC/Ni-Al2O3 composites obtained by SHS

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Combustion synthesis commonly called SHS (for self-propagating high-temperature synthesis) is a promising method employed for the synthesis of many advanced materials, such as ceramics, composites, intermetallics, etc. This method is based on the use of the heat released during exothermic reactions in order to preheat raw materials and to obtain a self-sustained system.
The main advantages of the SHS technique are the high cost-effectiveness associated with the low energy requirement, short processing time, simplicity of facilities, and formation of high-purity products. However, the SHS composites obtained had poor mechanical properties due to the coarse-grained microstructure exhibited.
As a model system for SHS processes, the Ti-C system, has been widely studied. The heat generated by the reaction between titanium and carbon is large enough to sustain a combustion wave propagation, once the reaction is initiated. However, the reaction is extremely fast and the final product is highly porous. The addition of diluents is sometimes required to lowering combustion temperature and decreasing the reaction rate, which help achieve the appropriate microstructure.
In this study, 5 and 10 wt% of thermite mixture NiO-Al, were added to the stoichiometric mixture Ti-C to produce TiC-Ni-Al₂O₃ composites. The formation of a by-product alumina-nickel could be sought for the hardness of the final product, since Ni is known to be a good sintering aid for titanium carbide and the presence of Al₂O₃ is usually not a drawback as it is usually considered as a reinforcement.
to monitor in situ the structural and thermal evolutions taking place during the SHS reactions, a time-resolved X-ray diffraction (TRXRD) experiments using an X-ray synchrotron beam (ID11, ESRF Grenoble) was carry out with a short acquisition times (35ms/scan). This analysis enable us to observe several steps including intermediate phases' appearance-disappearance, aluminum melting and thermite reaction before obtaining the final product. Those results were then, compared with earlier study results on the Ti-C-Ni system, where the Ni was a part of the reactive mixture.
Tracking the evolution of the lattice parameters of TiC during its formation enabled us to estimate the critical temperatures for those SHS reactions.
Products, were analyzed by SEM observations.

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