THERMOMECHANICAL STUDY OF ALUMINO-SILICATE COMPOSITES

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Abstract: Layered alumino-silicate composites, used as refractories, are studied to determine their thermomechanical behavior through a thermal treatment up to 1550°C. Reactions in the matrix and matrix-particle interactions create a secondary mullitization (~24 wt.% increase of mullite amount) in the structure that consequently strengthens the material. Samples with higher pore volume fractions exhibit lower overall Young's modulus values.

Key words: Refractory, Mullite, Thermomechanical Properties.

1. INTRODUCTION

Alumina and silica, two of the most common compounds in earth's crust, have been widely used in refractory materials through history. Mullite is the only stable intermediate phase in the alumina–silica system at atmospheric pressure; it is mostly present in man-made ceramics, very rarely occuring spontaneously in nature. Many of its high temperature properties are superior to those of most other metal oxide compounds, including alumina. In the present work, refractory layered materials (composed by an alumina/silica matrix reinforced with mullite particles) are studied via high temperature tests to determine the effect of phase evolution during sintering on their thermomechanical behavior[1].

2. MAIN CHARACTERIZATION TECHNIQUES

2.1. Ultrasonic long bar echography (US-HT)

The ultrasonic long bar mode method at high temperature allows investigating the evolution of Young's modulus in dependence of temperature for the studied material (Fig.1.b). A magnetostrictive transducer linked to an alumina wave-guide emits an ultrasonic wave; this tension-compression ultrasound wave is transmitted to the sample in a furnace through the wave-guide (Fig.1.a)[2].

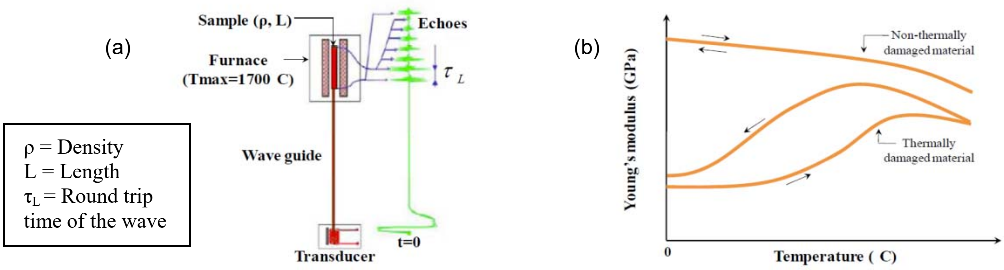


Fig.1. (a) Schematics of the ultrasonic long bar mode technique at high temperature. (b) Typical evolutions of Young's modulus in dependence of temperature[2].

2.2. Acoustic Emission (AE)

High temperature acoustic emission is based on the record of acoustic waves induced by energy release phenomena occurring within the material (damage) during external solicitations[3]. When a material is subjected to a stimulus (thermal path), localized initiators trigger the release of energy, in the form of stress waves, which propagate to the surface (damage) and are recorded by sensors (Fig.2).

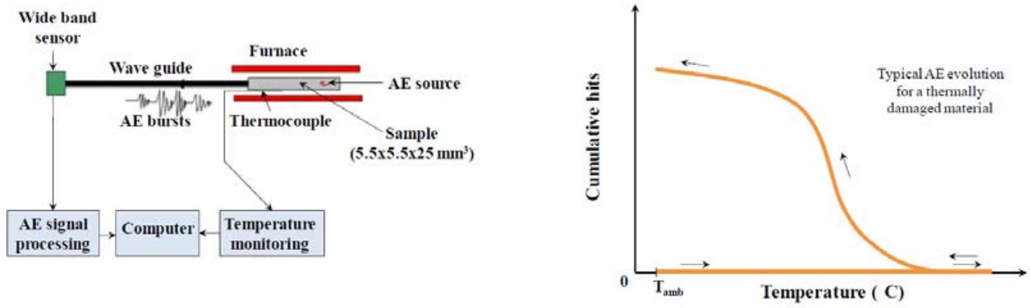


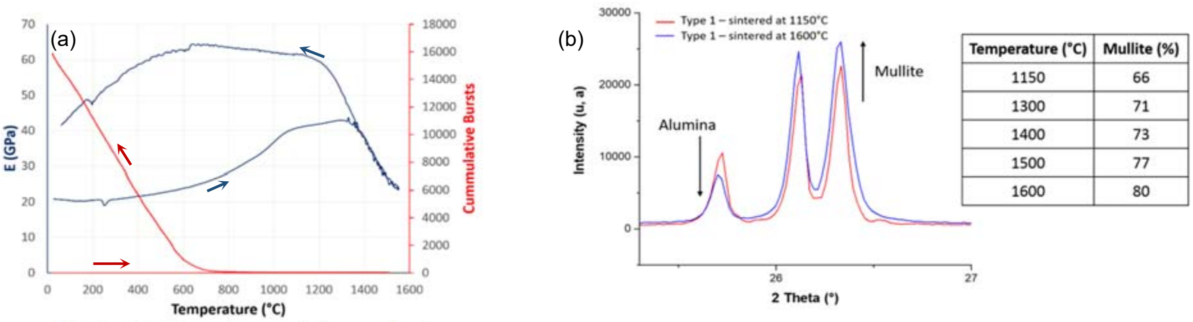
Fig.2. Schematics of the acoustic emission technique at high temperature, and the typical evolution for a thermally damaged material[2].

2.4. Additional Methods

Samples are also analyzed by Dilatometry, X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM).

3. RESULTS AND DISCUSSIONS

This study comprised samples with mainly two types of reinforcements: 1) dense and 2) hollow mullite aggregates both embedded in the same alumina/amorphous silica matrix. Thermomechanical properties and phase evolution are determined throughout a thermal cycle up to 1550°C. All samples exhibit mechanical strengthening with temperature increments (Fig.3.a); as it occurs in most alumino-silicate mixtures, in situ secondary mullitization might take place as a reaction between amorphous silica and alumina particles present in the matrix, as well as interactions with reinforcement particles. XRD analysis was done to observe the evolution of crystalline phases during each step of heat treatment, and through Rietveld Analysis, it was possible to quantify the increase in mullite proportion with temperature; thus justifying the rigidization previously observed (Fig.3.b). During cooling, a decrease of Young's modulus (E) indicates the start of microcracking in the structure, which is highlighted by the start cumulative bursts in AE tests (Fig.3.a).



Temperature (°C)	Mullite (%)
1150	66
1300	71
1400	73
1500	77
1600	80

Fig.3. (a) Young's modulus evolution and cummulated salves with temperature for Type 1 samples. (b) Evolution of mullite with temperature for Type 1 samples.

Type 2 specimens had an overall lower mechanical strength than Type 1 samples, caused by higher porosity.

4. CONCLUSIONS

Alumino-silicate composites undergoing high temperature heat treatments show an increase of Young's modulus caused by secondary mullitization in the matrix and between matrix-reinforcement particles.

REFERENCES

[1] Duval, D. Mullite - Ceramic and Glass Materials: Structure, Properties and Processing, Springer, 2008, 27-40.
[2] Grasset-Bourdel, R. Structure/property relations of magnesia-spinel refractories: experimental determination and simulation - Thése doctorale, 2011, Université de Limoges.
[3] Chotard, T. High temperature characterization of Cordierite-Mullite refractory by ultrasonic means. Journal of European Ceramic Society, 2008, 2129-2135.