CREEP BEHAVIOUR OF Al_2O_3 / Al_2O_3-FIBRES AND MULLITE / MULLITE-FIBRES MATERIALS OBTAINED BY SPARK PLASMA SINTERING

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INTRODUCTION

The lowest creep deformation at high temperatures published up to now was observed on non oxide materials as Si_3N_4 or alumina-SiC nanocomposites. However the main problem of non-oxide ceramics is oxidation, which strongly decreases their deformation resistance in air at high temperatures. In the present work, alumina and mullite based composites with up to 20vol% of alumina short fibres densified by using Spark Plasma sintering are proposed as prior candidates for high temperature creep resistant materials. The influence on the deformation behaviour of different fibre contents and sintering temperatures was studied. The results were compared with alumina nano-SiC nanocomposites in the temperature range from 1200 to 1400ºC. Al_2O_3 / Al_2O_3-fibres and Mullite / Al_2O_3-fibres composites presented both, very low creep rate and good thermal stability at high temperatures being promising materials for thermomechanical applications.

MATERIALS PROCESSING

Materials dispersion in boiling ethanol → Magnetic stirring at 100ºC (up to solvent evaporation) → Sieving → Spark Plasma Sintering → Alumina fibres composite

STARTING MATERIALS.

Alumina. Taimei TM-DAR

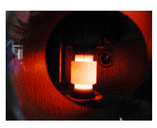
Mullite. Baikowski 193 CR.

Alumina fibres (A_F). Neoker

COMPOSITION.

5% vol. A_F

20% vol. A_F



Spark Plasma Sintering in vacuum at 80MPa

Why SPS?

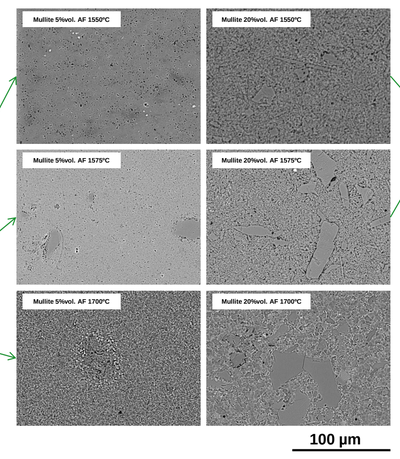
Densification enhancement.

Full dense fibre based materials

Effect of temperature

MULLITE A_F COMPOSITES

SAMPLE CODE	FIBRE CONTENT (%VOL)	SINTERING TEMPERATURE (ºC)	DENSITY (%)	
MAF-5 -1550	5	1550	92.9± 0.7	Lower eutectic point of Al2O3-SiO_2 system
MAF-20- 1550	20	1550	90.8 ± 0.1	
MAF-5- 1575	5	1575	97.9 ± 0. 1	Near eutectic point of Al2O3-SiO_2 system
MAF-20 -1575	20	1575	93.0 ±0.1	
MAF-5 -1700	5	1700	99.2 ± 0.2	Upper eutectic point of Al2O3-SiO_2 system
MAF20 1700	20	1700	99.4 ± 0.03	

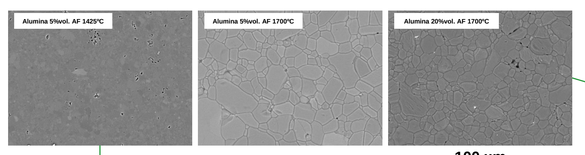


Porosity

FULL DENSE MATERIALS AT 1700ºC

ALUMINA A_F COMPOSITES

SAMPLE CODE	FIBRE CONTENT (%VOL.)	SINTERING TEMPERATURE (ºC)	DENSITY
AAF-5-1425	5	1425	98.2 ± 0.1
AAF-5-1700	5	1700	99.5 ± 0.1
AAF-20-1700	20	1700	99.5 ± 0.1



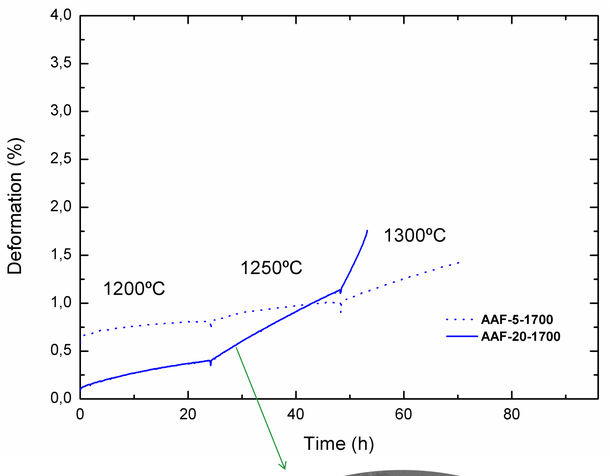
Niquel impurities coming from fibres

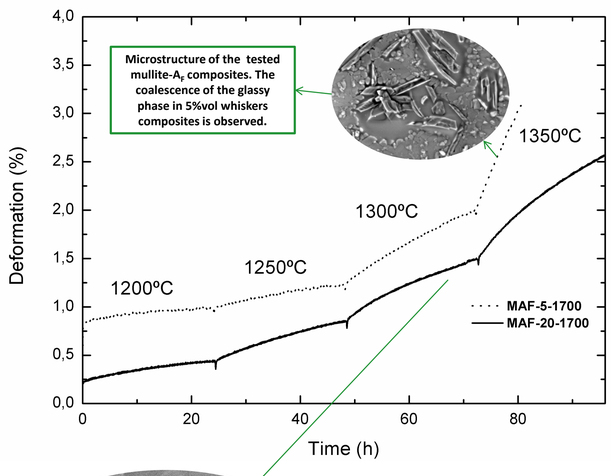
Porosity

HIGH TEMPERATURE BEHAVIOUR

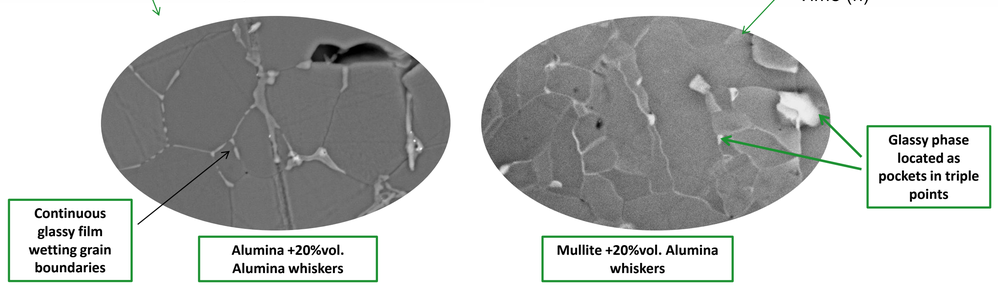
Deformation and creep rate of the final materials have been measured by means of three point bending tests. The range of temperatures was varied from 1200ºC to 1350ºC. For each sample the temperature was increased 50ºC every 24h. The load remained constant during the experiment and fixed at 100MPa. Only full dense materials have been tested (corresponding with those sintered at 1700ºC).

Effect of fibre content





Microstructure of the tested mullite-A_F composites. The coalescence of the glassy phase in 5%vol whiskers composites is observed.

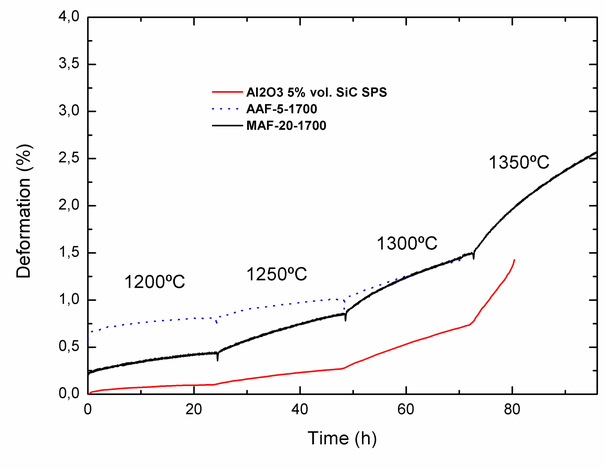


Mullite with higher whiskers contents presented better deformation resistance than alumina with the same alumina fibres content and SPS sintered at the same conditions. This fact is assumed to be due to the presence of glassy phase free grain boundaries in mullite

MULLITE MATERIALS WITH HIGHER FIBRE CONTENTS AND SINTERED BY SPS AT 1700ºC SHOWED THE BEST HIGH DEFORMATION RESISTANCE.

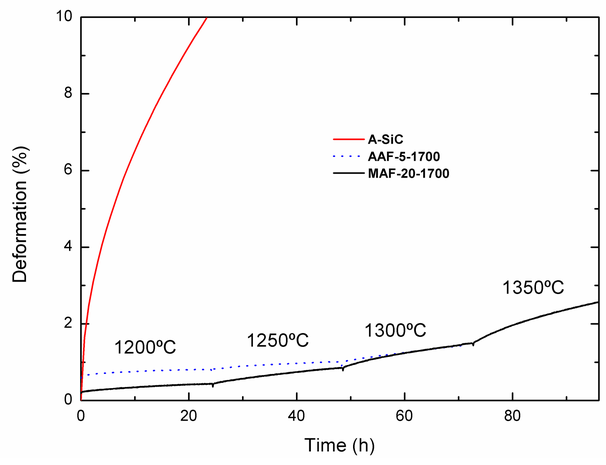
Comparison with non oxide ceramics

AS SINTERED



Alumina-SiC Materials presented power deformation when tested as sintered

TREATED IN AIR AT 1000ºC DURING 5H



When the material is slightly oxidized before testing creep resistance of alumina-SiC materials decreases in various orders of magnitude.

MULLITE-AF AND ALUMINA-AF MATERIALS REPRESENT A MORE RELIABLE OPTION FOR OXIDIZING CONDITIONS.

CONCLUSIONS

- A_F –mullite and A_F –alumina composites up to 20 vol% of fibres and densities up to 99% have been achieved by means of Spark Plasma Sintering.
- Creep resistance of the A_F –mullite and A_F –alumina composites sintered at the same conditions have been compared . The improvement in deformation resistance observed for mullite-whiskers materials has been explained by the absence of glass wetting the grain boundaries.
- Creep resistance of Mullite-A^F and Alumina-A^F has been compared with the resistance of and Al2O3-SiC (5%vol.) sintered in similar conditions. It has been demonstrated that when the material are in oxidizing media Mullite-A_F and Alumina-A_F presented a deformation considerably lower than alumina-SiC. According to that, A_F based material represent a more reliable option for thermomechanical application in air.

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REFERENCES

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