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and
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Advanced Ceramics and their Composits For Energy Application

Presented by :
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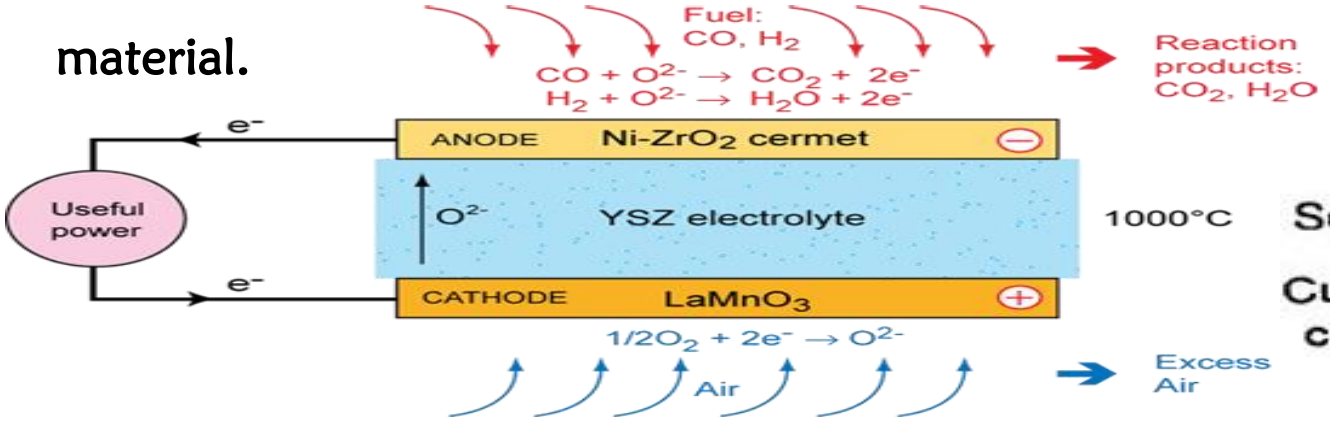
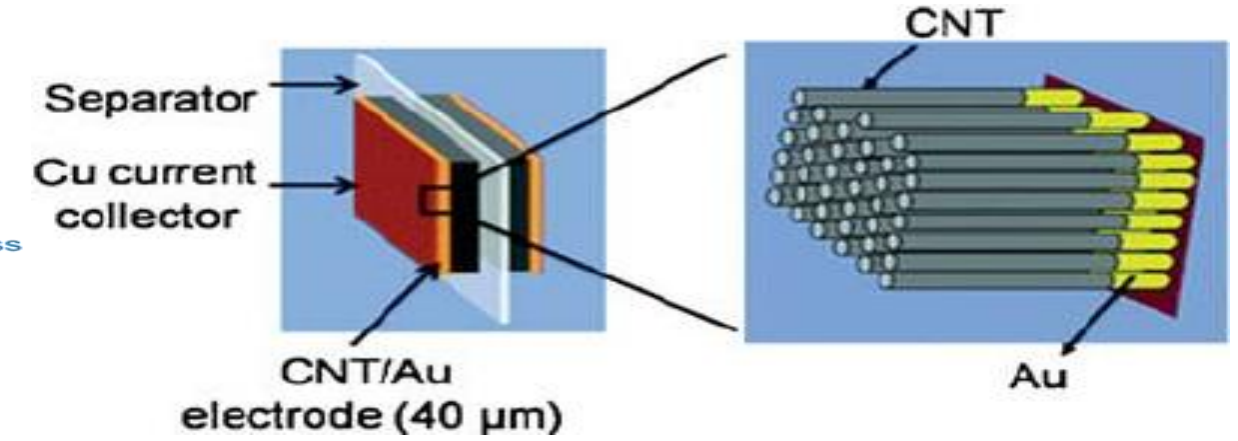
Content

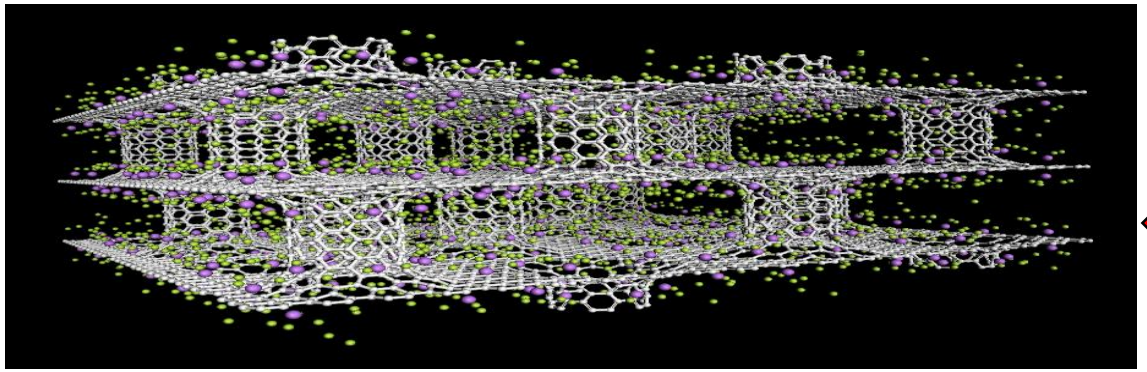
- General introduction of the thesis topic.
- Results and discussions.
- Conclusion and future perspectives.

❖Introduction

❖ Nowadays, the investigation of advanced ceramics, namely zirconia and their composites became an important task of research for tremendous scientific laboratories dealing with energy production, conversion and storage. The incorporation of a second phase such as MWCNT into zirconia matrix appears as a very promising technology, especially for enhancing the thermo-mechanical and the electrical properties of the material.







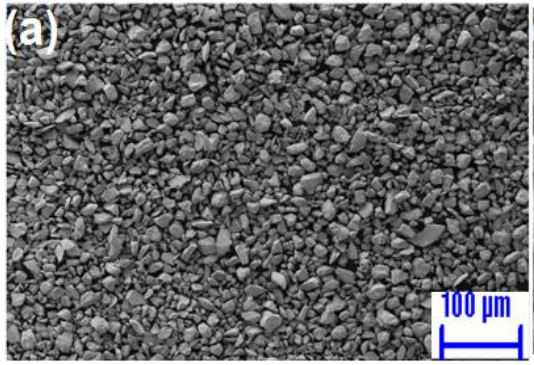
Modern Hydrogen Storage material

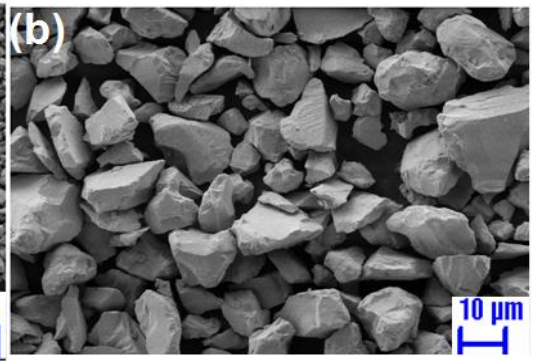
❖Powder preparation

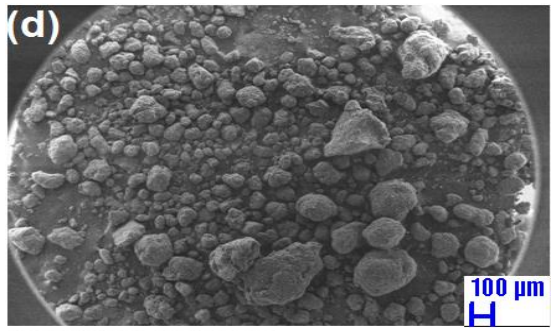
❖The first step of our research topic consisted mainly on investigating the effect of attritor milling on the structural and thermo-mechanical properties of 8 mol% yttria- stabilized zirconia (8YSZ) composites with 1wt%, 5 wt% and 10 wt% MWCNTs addition. The composites were milled at 4000 rpm in ethanol for 5 hours using an attritor mill. The influence of MWCNT content on the microstructure were assessed by scanning electron microscope (SEM), (HRTEM). and energy dispersive X-ray spectroscopy (EDS), (XRD), and RAMAN spectroscopy.

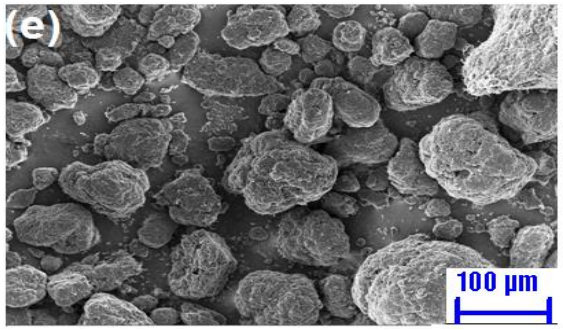
Sample notation	ZrO_2 (wt%)	MWCNT (wt%)	Milling ethanol (4000 rpm)
A	100	0	5 h
B	99	1	5 h
C	95	5	5 h
D	90	10	5 h

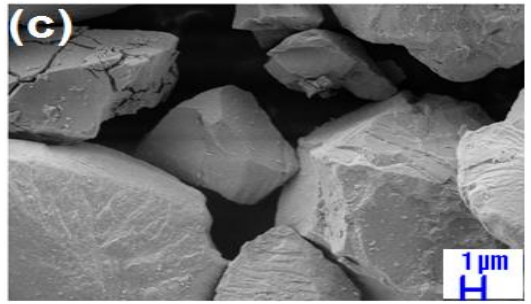
❖Structural investigations of composites

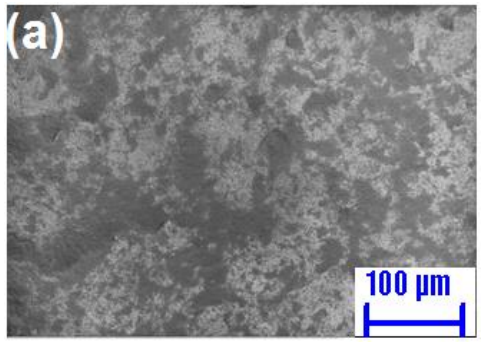


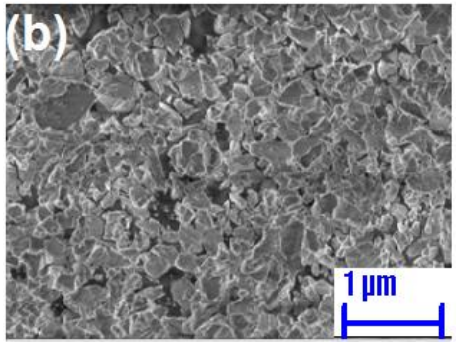


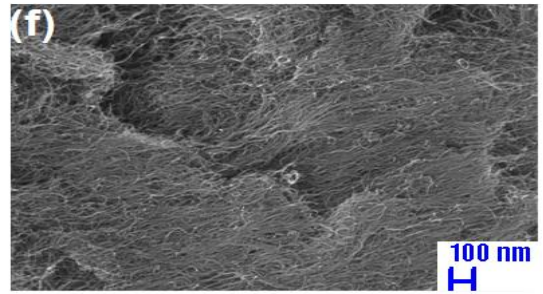






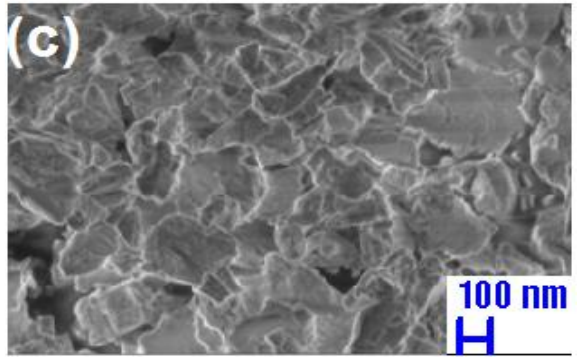






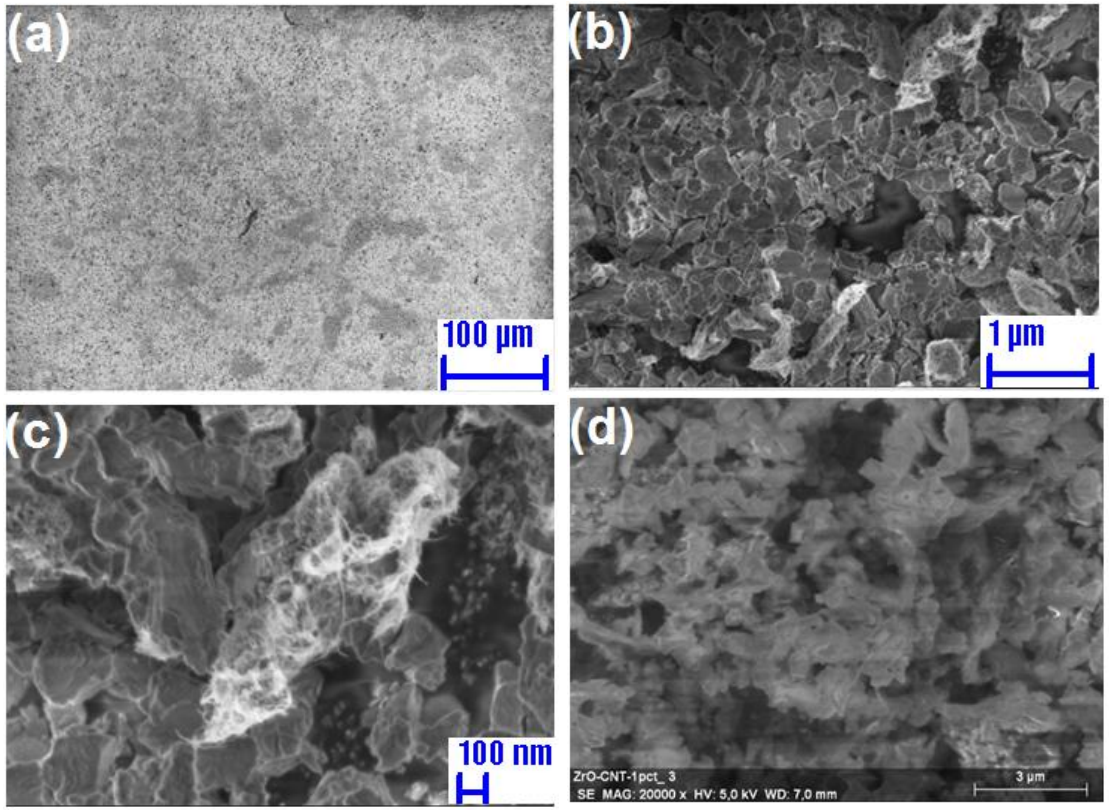
❖As received zirconia exhibited a well dispersed particle with a sharp and irregular shape. The average size of the grains was limited between 40 and 100µm.

❖As received MWCNT clustered as bundles of 2.5µm in length. The bundles contain a huge network of MWCNT, each nanotube has a relative number of layer variating from 7 to 9.

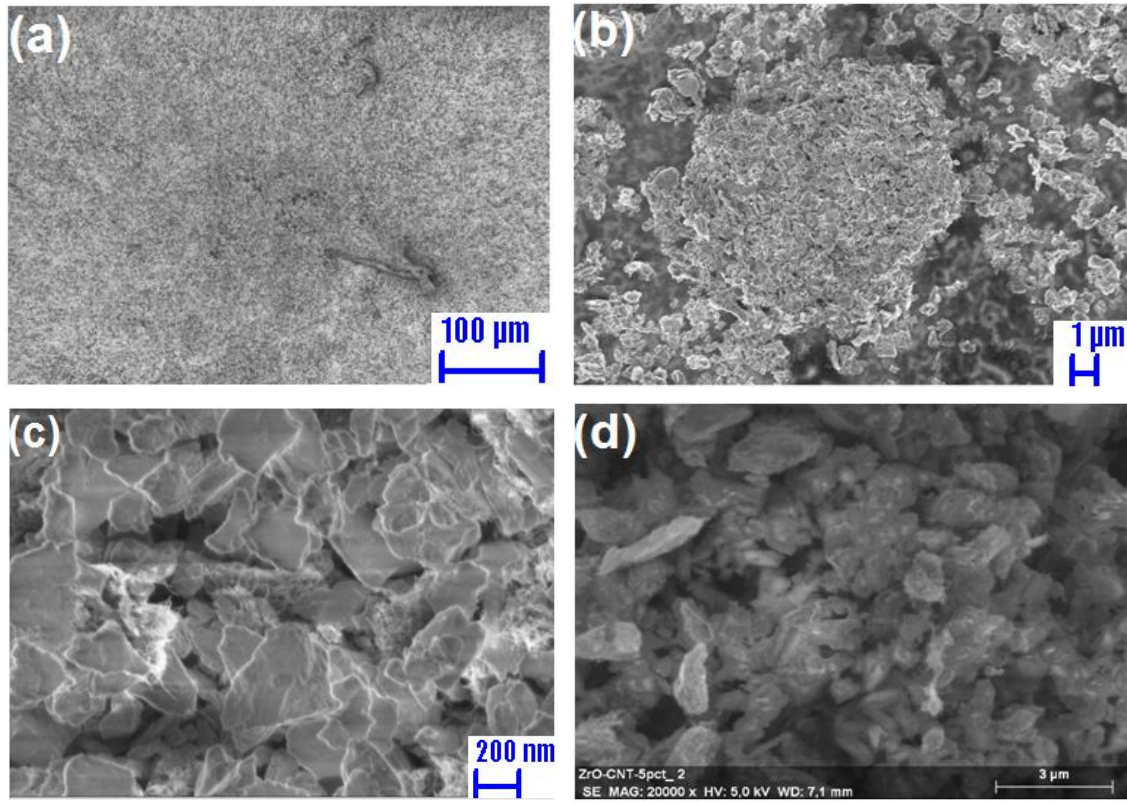


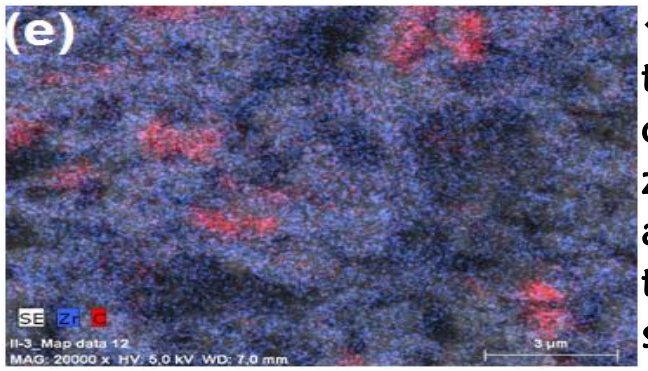
❖Sample A showing more homogeneous distribution and a smaller size of the grain from 200 to 300 nm after intensive milling compared to the as received material.

❖YSZ with 1 wt% of MWCNT content

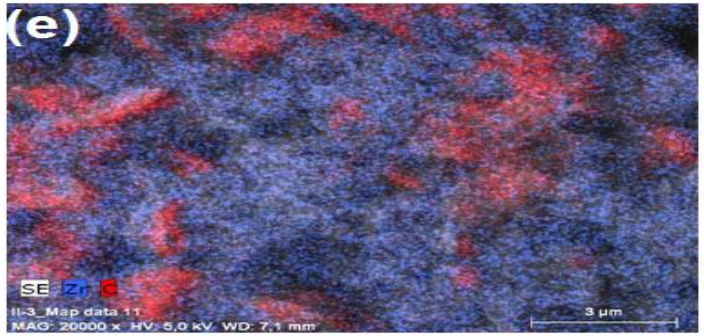


❖YSZ with 5 wt% of MWCNT content

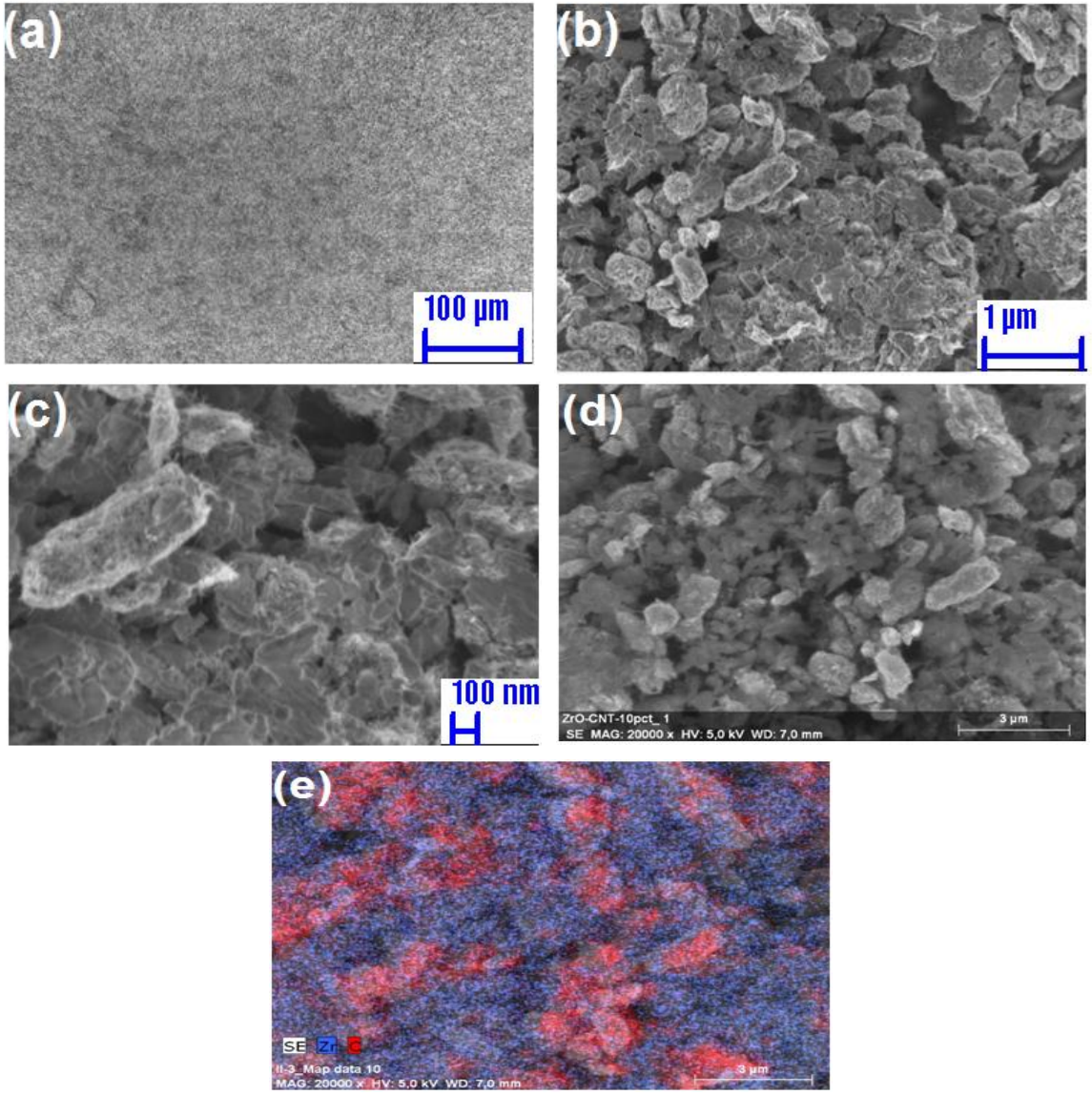




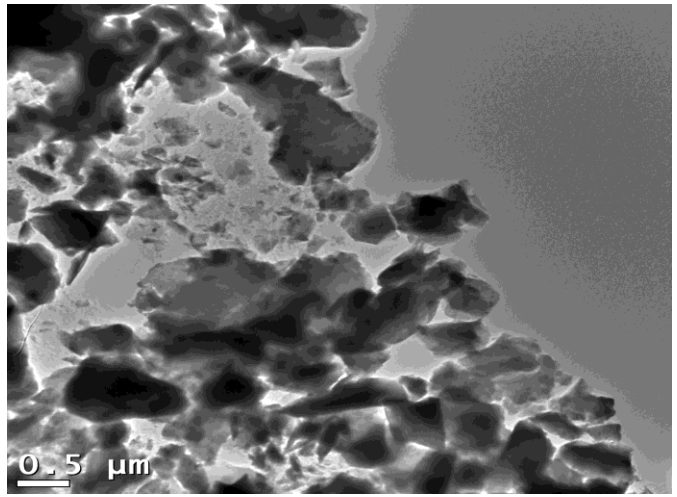
❖The grains change the surface topology and exhibit various dimensions with adding MWCNT to zirconia matrix. The agglomeration has a dark phase increasing from sample C to sample D. While, zirconia grains surrounding theses agglomerations, show a significant refinement compared to the reference.

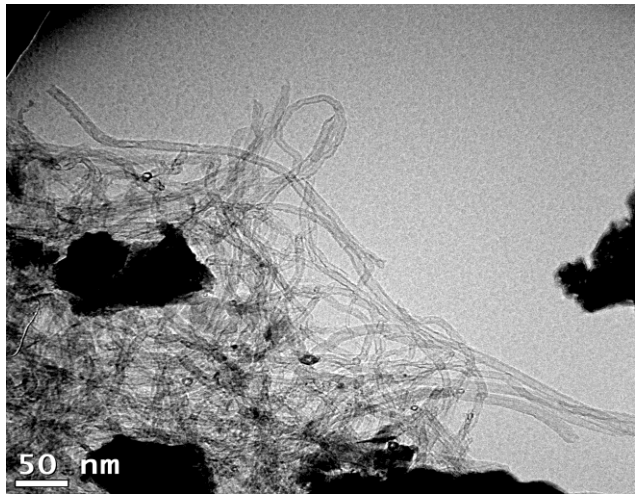


❖YSZ with 10 wt% of MWCNT content.



High resolution TEM images for as-received MWCNT indicated an approximative outer/inner diameter value of 9,13nm and 3,8nm respectively.

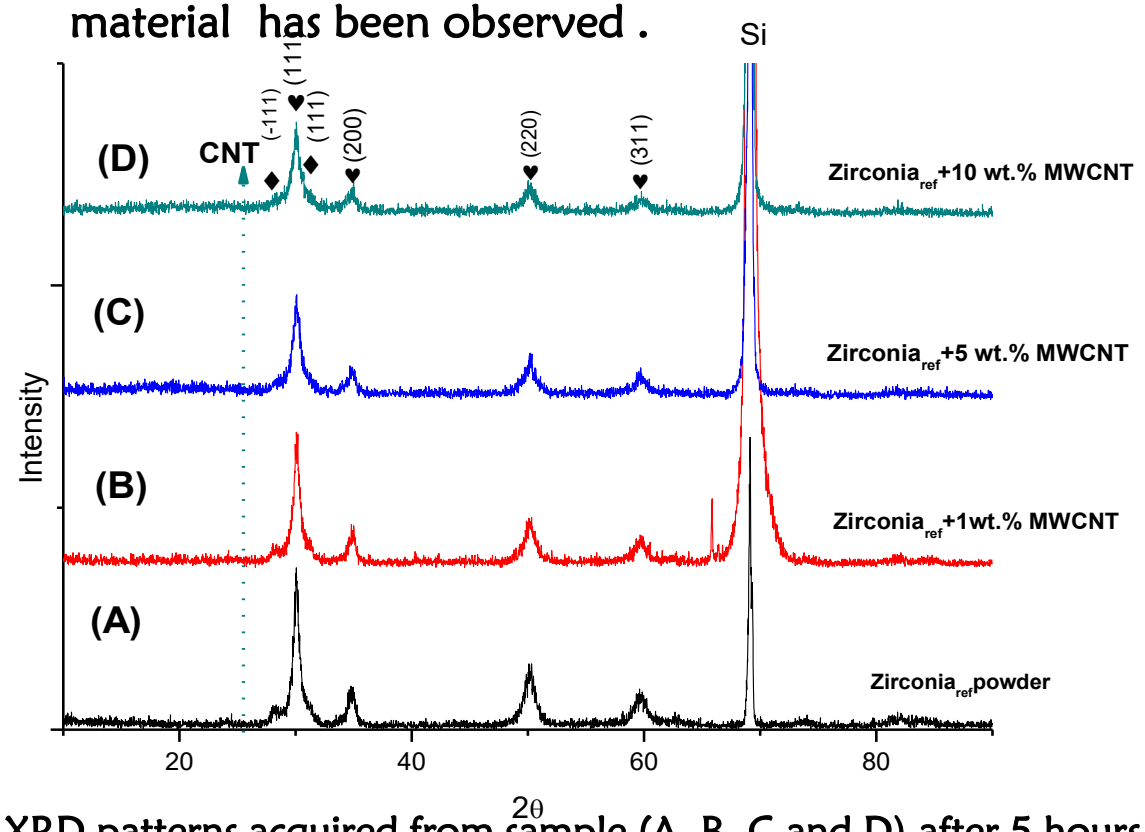




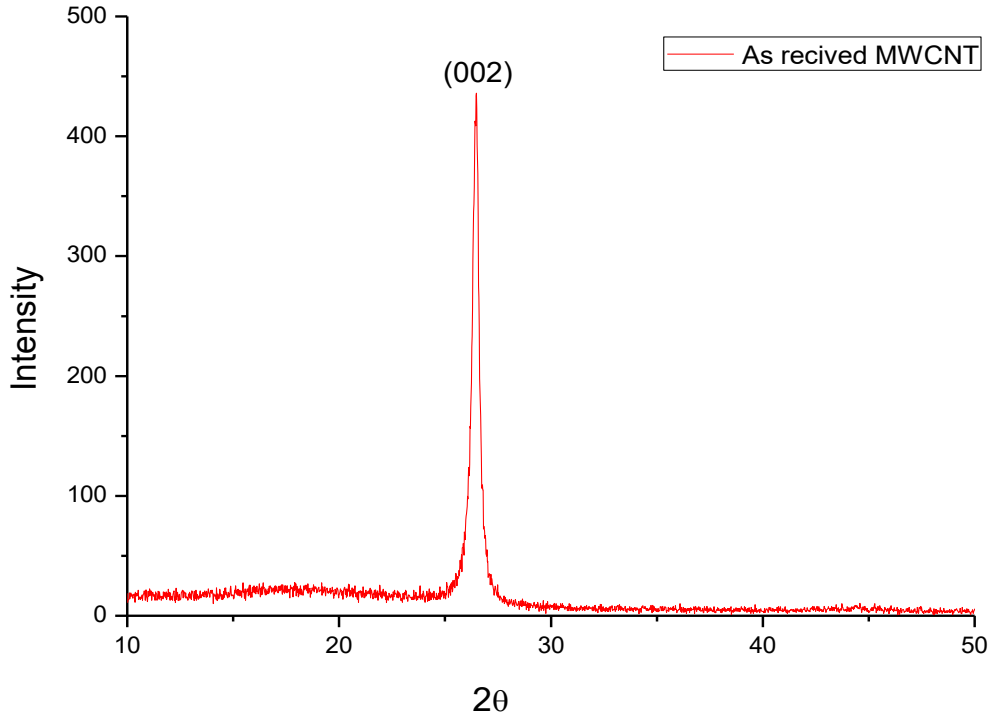
High resolution TEM images for sample C indicating the dimension of the smallest and the largest grain about 50nm and 1.5µm respectively.

❖XRD investigation :

❖The XRD patterns revealed that all the composits were composed mainly by a cubic zirconia indicated by lines at 2Θ of 30.24°, 34.88°, 50.17°, 59.69° , a small fraction of monoclinic zirconia was observed as well at at 2Θ of 28.36° and 31.12°. A significant decrease in the intensity of the cubic and the monoclinic zirconia lines with MWCNT addition, besides the absence of line at 2Θ of 26° , which is a characteristic of the graphitic material has been observed .



XRD patterns acquired from sample (A, B ,C and D) after 5 hours of milling.



XRD patterns of as received MWCNT showing the appearance of peak at 2Θ of 26°.

❖Raman spectra investigation :

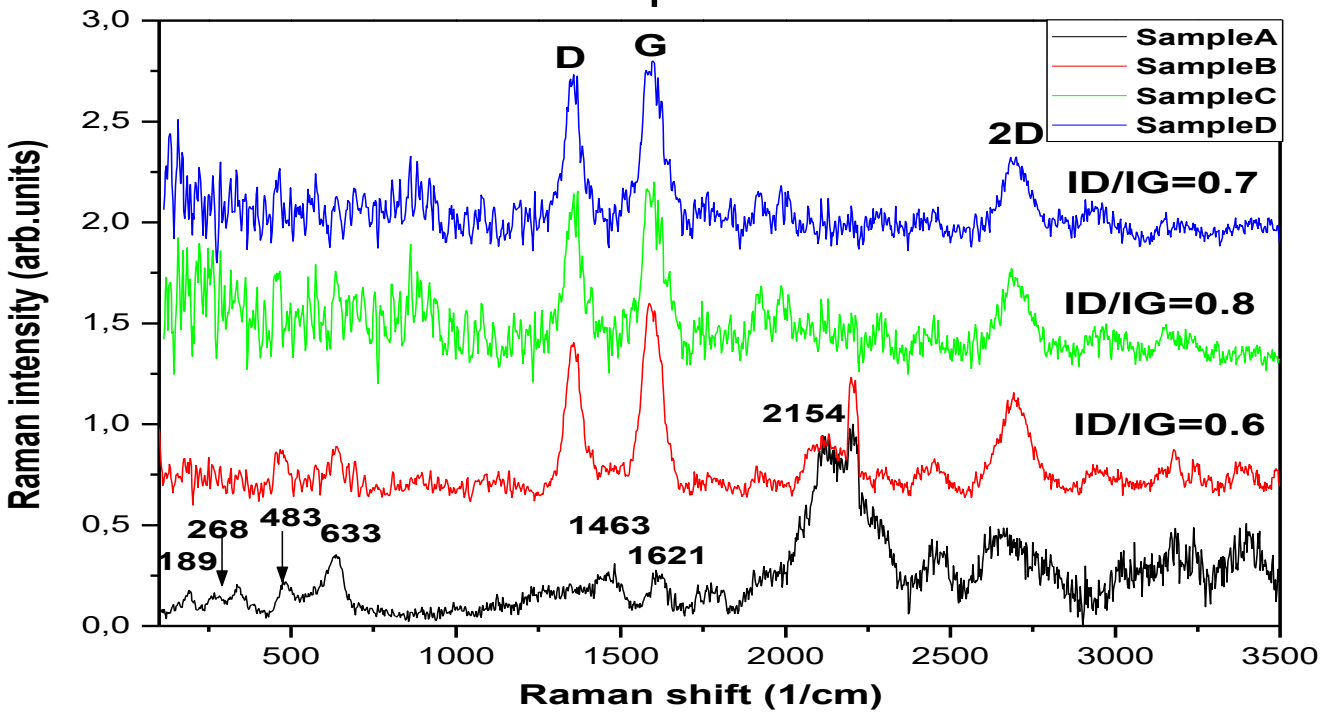
Observation:

❖The Raman shift from 0 to 1000 cm^{-1} (sample A) indicate the presence of zirconia peaks at 187cm^{-1}, 274cm^{-1}, 341cm^{-1}, 380cm^{-1}, 475cm^{-1}, 637cm^{-1}.

❖Similarly RAMAN spectra presents the apparition of the G and D bands for all the composites at an average Raman shift of ~1589cm^{-1} and ~ 1356cm^{-1} respectively.

Interpretation of the results:

❖These results could be interpreted considering different factors: The inhomogeneity of the mixture or due to the small examinates spot around 1µm from the total area of the sample.

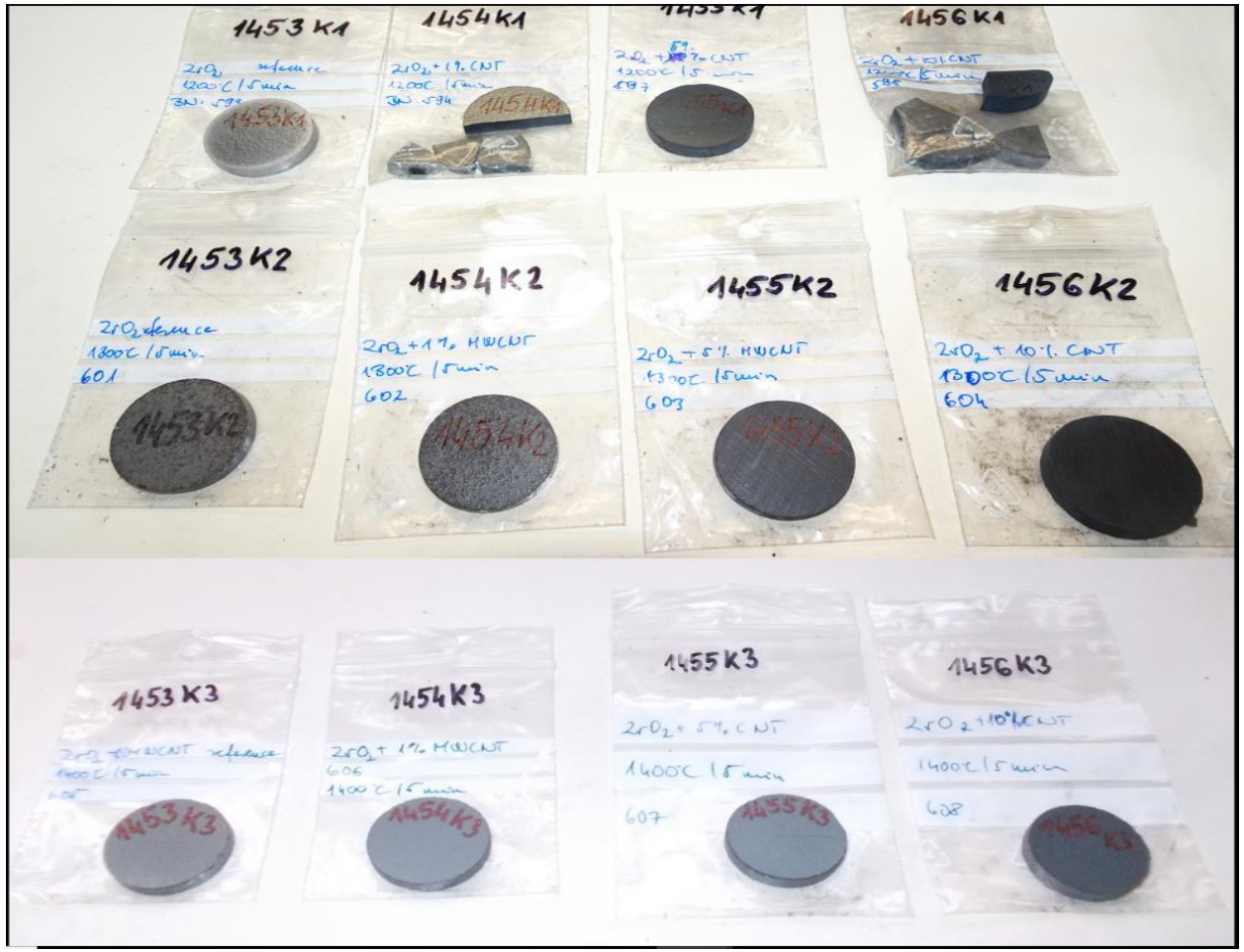


Raman spectra using a laser excitation wavelength at 488nm for sample(A , B , C, and D).

❖Sintering treatment

Type of the sample	Number of packet	Available amount of powders/g. packet	The necessary amount of sintering for each sample	Dimension of the samples (Diameter , height)	First sintering caracteristiques	Second sintering caracteristiques	Third sintering caracteristiques
Reference sample :1453 Cubic Zirconia	3	First packet :21.5 g Second packet: 21.5 g Third packet: 21.5 g	20g	(30 mm , 5 mm)	Temperature: 1200◦C Time:5 min Pression:60 MPa heating and cooling rates:200◦C/min	Temperature: 1300◦C Time: 5 min Pression:60MPa heating and cooling rates:200◦C/min	Temperature: 1400◦C Time: 5 min Pression:60MPa heating and cooling rates:200◦C/min
1454 Cubic zirconia + 1wt% MWCNT:	3	First packet : 21.5 g Second packet: 21.5 g Third packet: 21.5 g	20g	(30 mm , 5 mm)	Temperature: 1200◦C Time:5 min Pression:60 MPa heating and cooling rates:200◦C/min	Temperature: 1300◦C Time: 5 min Pression:60MPa heating and cooling rates:200◦C/min	Temperature: 1400◦C Time: 5 min Pression:60MPa heating and cooling rates:200◦C/min
1455 Cubic zirconia + 5 wt% MWCNT:	3	First packet : 21.5 g Second packet: 21.5 g Third packet: 21.5 g	20g	(30 mm , 5 mm)	Temperature: 1200◦C Time:5 min Pression:60 MPa heating and cooling rates:200◦C/min	Temperature: 1300◦C Time: 5 min Pression:60MPa heating and cooling rates:200◦C/min	Temperature: 1400◦C Time: 5 min Pression:60MPa heating and cooling rates:200◦C/min
1456 Cubic zirconia + 10 wt% MWCNT:	3	First packet : 21.5 g Second packet: 21.5 g Third packet: 21.5 g	20g	(30 mm , 5 mm)	Temperature: 1200◦C Time:5 min Pression:60 MPa heating and cooling rates:200◦C/min	Temperature: 1300◦C Time: 5 min Pression:60MPa heating and cooling rates:200◦C/min	Temperature: 1400◦C Time: 5 min Pression:60MPa heating and cooling rates:200◦C/min

❖Reference samples and composites of zirconia with 1wt%, 5 wt% and 10 wt% MWCNTs addition, sintered at 1200◦C, 1300◦C, 1400◦C



Different treatments of the sintered samples

Characterization of the material

- Raman SEM XRD TEM measurement after and before sintering for all the samples.
- Analyse the effect of the MWCNTs addition and the sintering temperature on the microstructure and the mecanoelectrical properties of the samples.
- Raman SEM XRD TEM measurement after and before the three point bending.

Experimental measurement

- Grinding and polishing
- Three point bending
- Hardness
- Electrical conductivity

❖Conclusion and perspectives

❖As a further perspective, we are looking to extend the result of this work after sintering treatment and suggest a new material with a better mechanical and electrical properties applicable for energy storage or power generation systems.

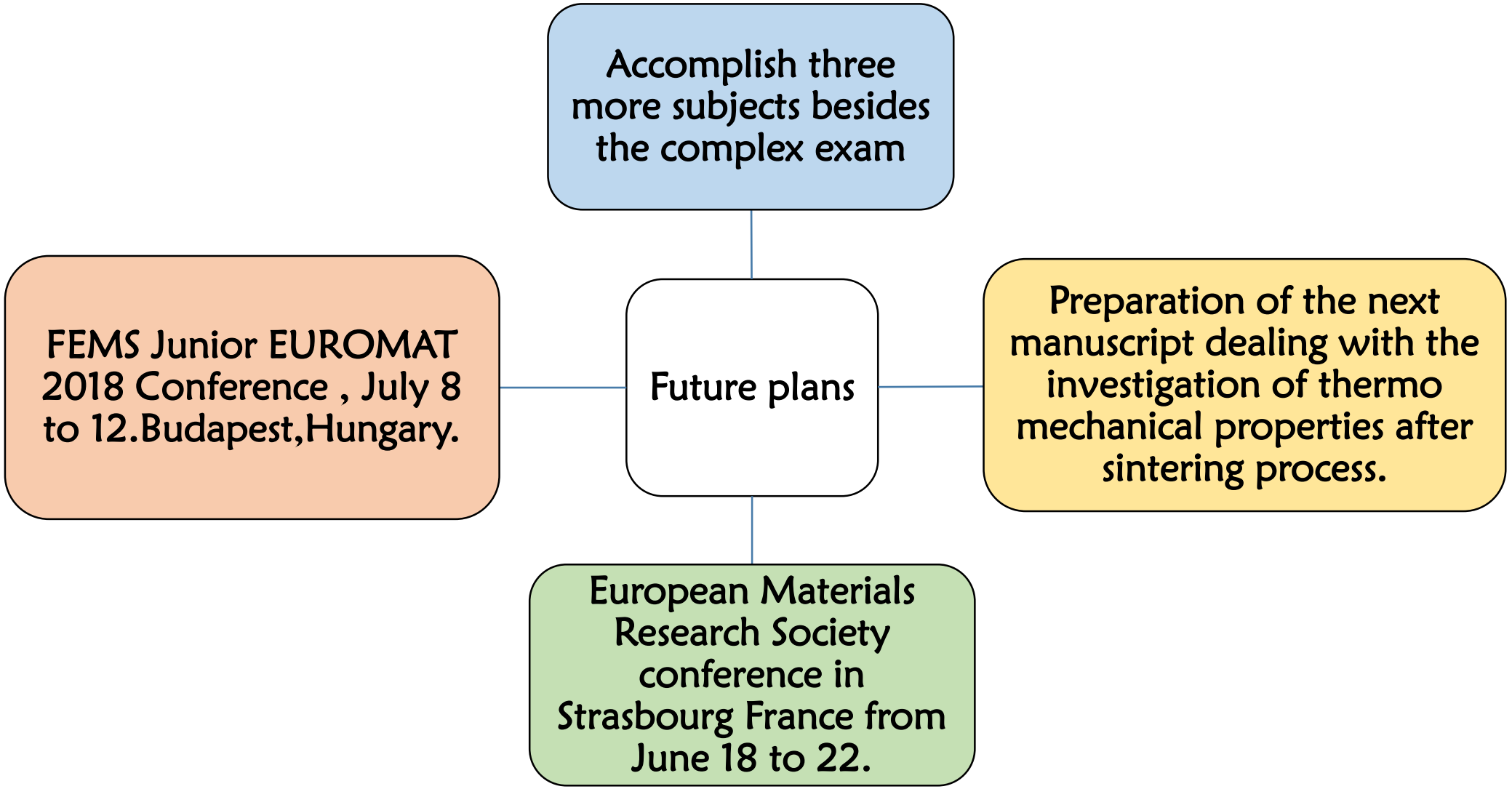
❖*Poster 1*: S. Lamnini, K. Balázsi, Cs. Balázsi, Advanced ceramic and their composites based on zirconia for energy application, ECerS 2017, 15th Conference & Exhibition of the European Ceramic Society, 2017. Július 7-13, Budapest, poster

❖*Poster 2:* S. Lamnini, K. Balázsi, Cs. Balázsi, Advanced ceramic and their composites based on zirconia for energy application, International Conference - Deformation and Fracture in PM Materials, High Tatras, 2017. Okt.22-25, poster

❖*Laboratory work*.

❖*Article untitled* : ***« EFFECT OF ATTRITOR MILLING ON GRAIN SIZE AND DISTRIBUTION OF CARBON NANOTUBES IN YSZ/CNT COMPOSITES ».*** *Under preparation*

❖**Courses :**Chemistry and physics of polymers and Ceramic .


Accomplish three more subjects besides the complex exam
FEMS Junior EUROMAT 2018 Conference , July 8 to 12.Budapest,Hungary.
Future plans
Preparation of the next manuscript dealing with the investigation of thermo mechanical properties after sintering process.
European Materials Research Society conference in Strasbourg France from June 18 to 22.

Köszönöm a figyelmet!

Thank you for your attention!