


# Portuguese Society for Microscopy

## SPMicros

*Fostering microscopy research since 1966*

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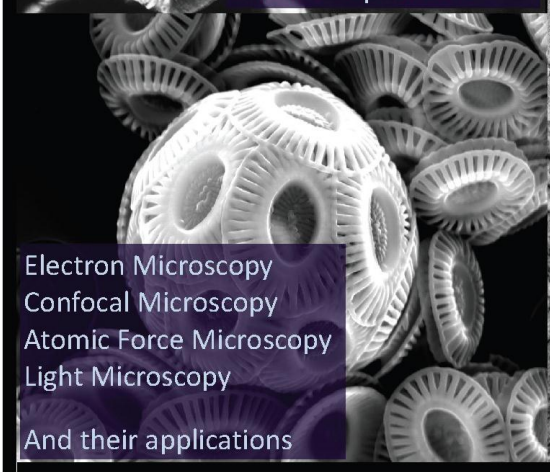

65 anual meetings and Joint Iberian Meetings with SME



Official journal: Microscopy and Microanalysis



Seminars and courses on microscopy methods and their applications



Electron Microscopy  
Confocal Microscopy  
Atomic Force Microscopy  
Light Microscopy  
And their applications



Life Sciences and Materials Science sections

## Welcome note

The Portuguese Society for Microscopy (SPMicros) has the pleasure of welcoming you to the 46th Annual Congress of the Society in the D. Estefânia Hospital in Lisbon.

The Congress brings together, in a multidisciplinary context, all colleagues who use microscopy techniques in their research or work. The technological focus of the Congress is fully justified by the recent advances in the microscopy fields, which are covered in plenary and keynote presentations, as well as in the associated Microscopy Course that has raised widespread interest.

SPMicros is focused on serving its members and the Annual Congress, in which ongoing work can be discussed with interested colleagues in an informal and friendly environment is one of the key activities of the society.

By conveying information and training useful for all, particularly for students who are initiating their contact with microscopy and all its potential, the Congress and associated Course, set the stage for an effective support to the needs of both junior and senior scientists and technicians looking for training and up to date information on the advancements in the field.

The SPMicros Board of Directors wishes to all participants a pleasant and fruitful congress and wishes to thank warmly to all collaborators that have struggled to solve the many challenges of the organization.

On behalf of the board of Directors,

António Pedro Alves de Matos  
President of SPMicros

## Characterization of $\text{Cu}_2\text{ZnSn}(\text{SSe})_4$ monograin powders by FE-SEM

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The design and synthesis of high-efficiency materials to convert solar to electrical energy is an increasingly important research field. Within the photovoltaic technologies, crystalline Si have an 80% share while the remaining 20% are mostly thin film solar cells based on  $\text{Cu}(\text{In,Ga})(\text{S,Se})_2$  (CIGSSe) and CdTe [1,2]. However, we must keep in mind the cost, abundance and environmental impact of the elemental components. For these reasons,  $\text{Cu}_2\text{ZnSnS}_4$  (CZTS),  $\text{Cu}_2\text{ZnSnSe}_4$  (CZTSe) and their solid solutions CZTSSe has attracted much attention recently and may provide a path to cost competitive solar cells. They consist of earth abundant and relatively inexpensive elements and represent an environmentally friendly alternative compared to the above mentioned systems [3]. The energy conversion efficiency of CZTSSe based solar cells has increased from 0.66% in 1996 to close to 10% recently [2].

The present work shows preliminary results that are related to the characterization of CZTSSe monograin powders by scanning electron microscopy. High purity metal compounds, S and Se powders were used as precursors for the synthesis of the  $\text{Cu}_2\text{ZnSn}(\text{SSe})_4$  monograin powders. The precursor powders were mixed to the desired composition and, additionally, KI was added as a flux material. Afterwards, the powders were blended in a mixer and encapsulated in quartz ampoules. The blended powders were degassed under dynamic vacuum at room temperature, sealed and annealed isothermally between 700 °C and 780 °C for a time ranging between 44 h and 136 h. After synthesis the flux material was removed with deionized water and the powders were sieved into several fractions. The morphology, microstructure and chemical composition of the synthesized powders was obtained with a Philips XL30 field-emission scanning electron microscope (FE-SEM) equipped with a backscattered electron (BSE) detector and an integrated EDAX energy dispersive X-ray spectroscopy (EDS) microanalysis system.

The typical morphology obtained for the CZTSSe powders can be seen in Fig. 1. Basically, the particles show a polyhedral morphology with some of them showing a needle shape, i.e. a large shape factor ( $L/D \gg 1$ ). Due to the complexity of the CZTSSe compound, it often contains other binary, ternary and quaternary phases and it is difficult to control the stoichiometry. Thus, it requires a very good control over synthesis parameters in order to obtain the desired phase of the material. SEM/BSE observations and EDS analysis are two powerful techniques to be used for this purpose. Fig. 2 puts in evidence the degree of the homogeneity of the CZTSSe monograins in relation to their size. The presence of undesired phases occurred more frequently in the larger powder particles ( $> 100 \mu\text{m}$ ).

1. Chung I., Lee B., He J., Chang R.P H., Kanatzidis M.G. (2012) Nature 485, 486-490.

2. Mitzi D.B., Gunawan O., Todorov T.K., Wang K., Guha S. (2011) Sol. Energ. Mater. Sol. Cell. 95, 1421–1436.
3. Muska K., Kauk M., Altosaar M., Pilvet M., Grossberg M., Volobujeva O. (2011) Energy Procedia 10, 203-207.

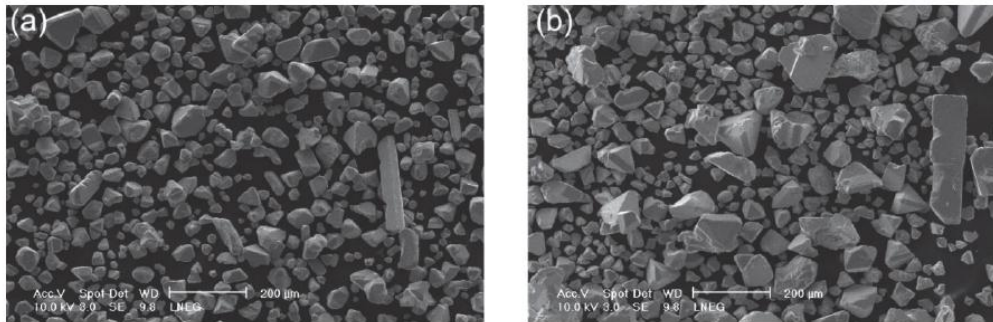


Fig. 1 - SEM images showing the typical morphology of the CZTSSe powders synthesized at (a) 780 °C/44 h and (b) 780 °C/ 136 h.

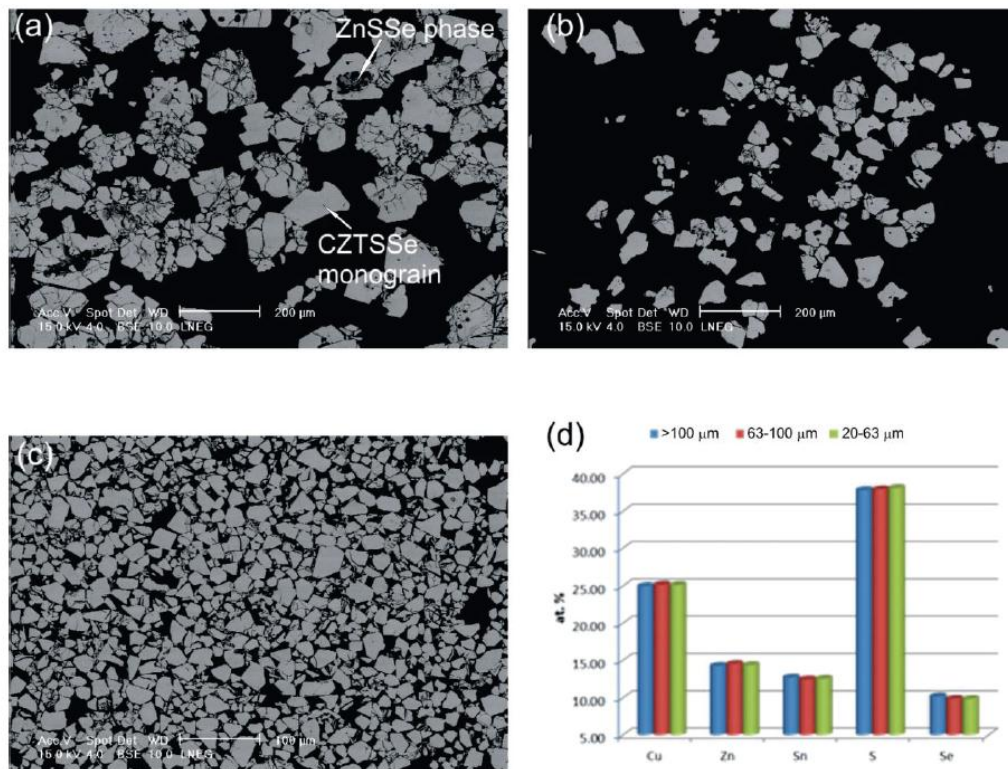


Fig. 2 - SEM/BSE images showing the typical microstructure of the CZTSSe powders synthesized at 700 °C/ 136 h, corresponding to different sieved fractions: (a) > 100 μm, (b) 63-100 μm, (c) 20-63 μm. (d) EDS semi-quantitative microanalysis results of the monograin powders.