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Assessing the homogeneity of an alumina/zircon powder mixture at various scales by scanning electron microscopy

Martin GIRAUD^{1,2}, Stéphane VAUDEZ², Cendrine GATUMEL¹, Guillaume BERNARD-GRANGER², Henri BERTHIAUX¹

¹ : Université de Toulouse, IMT Mines Albi, UMR CNRS 5302, RAPSODEE, Campus Jarlard, F-81013, Albi, France

² : ¹ CEA, DEN, MAR, DMRC, SFMA, LFC, F-30207 Bagnols-sur-Cèze Cedex, France

Powder mixing is a fundamentally important unit operation in industries handling powders. The purpose of such an operation is to reach a given homogeneity, which has to be defined at a certain scale, the observation scale, in order to be relevant. This scale is defined by the final product's requirements. Moreover, the homogeneity of a mixture can hardly be known exactly, and its assessment is always a scientific issue due to theoretical and practical limitations.

The most obvious limitation comes from the fact that it is rarely possible to get an exhaustive analysis of the mixture, since most analysis techniques require sampling. Thus, homogeneity has generally to be assessed from a restrained number of samples using statistical considerations. Finally, the analysis technique must enable a good differentiation of the powders while fulfilling the sampling procedure's requirements, specifically in terms of size and number of the samples.

In this study, alumina (Al_2O_3) and zircon (ZrO_2) powders have been used as simulants for an industrial process combining mixing and ball milling steps. Homogeneity of the blend among the whole process is crucial. Various binary mixtures of alumina (Al_2O_3) and zircon (ZrO_2) powders have been prepared using two different techniques: in a Turbula® mixer and in a dry ball mill. Within the ball mill, a reduction of the particle's size takes place simultaneously with the mixing operation. This allows homogenization at a much finer scale. Therefore, the knowledge of the homogeneity of the mixture at different scales is needed in order to identify the influence of different operational parameters on the homogenization of the mixtures during both processes.

The sampling was performed by drawing several portions, around two grams, into the final blends obtained by the Turbula® mixer and the ball mill. Each sample was then shaped into pellets in order to allow Scanning Electrons Microscopy observations without disrupting the samples. SEM has been used to perform sub-sampling of the pellets. Indeed, the back-scattered electrons signal analysis allows a very good differentiation of alumina and zircon due to their different atomic numbers. This technique also allows assessing the homogeneity of the mixtures at various scales by varying the magnification, and thus varying the size of the sub-samples. It is then relevant for both mixing paths.

Then, a MatLab® program has been developed in order to perform image analysis by thresholding and binarizing the SEM images. The program was also designed to perform statistical treatment of the samples and sub-samples providing sampling procedure for both scales. In particular, the number and size of the samples and sub-samples needed was computed.

Finally, this study provides a whole methodology to assess homogeneity of alumina and zircon mixtures at various scales for Turbula® mixing and ball milling.