Real-time material's response to high power microwave irradiation revealed by in-situ synchrotron radiation methods

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The complexity of microwave heating stems to a large extent from the intrinsic complexity of the materials exposed to microwave irradiation. For simple ideal homogeneous material, its size, shape and orientation with respect to the electromagnetic field influence the microwave absorption properties at the macroscopic scale. Furthermore, a number of material parameters, namely the complex permittivity and permeability, electrical conductivity, density etc. additionally determine the efficiency of the energy transfer from the microwave field to the sample. These material parameters are inter-correlated and may change significantly as function of frequency and temperature. For an homogeneous solid, the material parameters are defined by scalar values, i.e. having the same value throughout the specimen volume.

In multiphase and composite materials, the density and chemical constitution differs between the constituent phases. The material parameters responsible for the efficiency of microwave absorption can no longer be described by a small set of scalar values. Instead, the spatial variation of the magneto-dielectric properties, density, chemistry and degree of crystalline perfection is expressed in terms of scalar fields defined across all regions of nonzero density. Consequently, the microwave absorption efficiency and therefore the microwave heating capability of heterogeneous materials are determined by the local material properties at any point. The macroscopic material properties can be thought as statistical combinations of properties of numerous microscale homogeneous regions that constitute the specimen. As such, microwave heating is strongly sensitive to the microscopic details of the specimen, i.e. to its microstructure, density, chemical composition, crystal structure, strain-state, density and type of defects, granularity, aggregation, presence of surface oxide layers etc. Moreover, the above complex picture of microwave heating is not static. The close inter--relationship between the local microwave absorption properties, electrical conductivity and microscopic state parameters results in a fluctuating temperature field T(r,t), where r = (x,y,z) is an arbitrary position within the specimen. Local temperature gradients enhance diffusion and are occasionally generate hot-spots. Our ability to model and predict the complex interaction of heterogeneous materials with microwave fields is limited by (i) insufficient knowledge on the physics and chemistry of microwave absorption mechanisms, in particular when separate E/H fields, and (ii) the difficulty to follow in a time-resolved manner the rapid changes in the mass density distribution d(r,t) during microwave heating.

In this paper, we describe progress achieved at the Swiss Federal Laboratories for Materials Science and Technology (Empa, Thun, Switzerland) with respect to the real-time monitoring of microwave heating using in-situ X-ray microtomography and synchrotron radiation powder diffraction. Examples include non-equilibrium microwave heating and sintering of Al and ZnO powders, in-situ microwave infiltration processing and reactive microwave processing of metal-ceramic composites.