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Direct microwave sintering of gamma–alumina powder: Effect of alpha seeding and magnesia doping

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Microwave sintering as a rapid processing technic has been extensively investigated in the past decades. During microwave heating, the material couples with the electric field, absorbs the electric energy in its bulk and transforms it into heat. The microwave coupling behaviour depends on the dielectric loss factor. In most of pure oxide ceramics such as alumina the loss factor is very low at room temperature and increases with increasing temperature. Routinely a high dielectric loss material is used as a susceptor for heating the green specimen up to a temperature at which it significantly couples with the microwaves ; this process is referred to as “hybrid microwave sintering”.

The dielectric properties of alumina strongly depend on doping elements such as magnesium and yttrium or non-controlled impurities, especially in the microwave frequency range. In the literature, most studies deal with doped powders sintered in a multimode cavity using a susceptor. Spectacular effects of microwaves on densification and grain size were often reported and claimed. However, in most cases, the heating cycles in conventional sintering and microwave were different and the specimen temperature in microwave sintering could have been underestimated due to unreliable temperature measurement. In almost all cases, the authors unsuccessfully tried direct microwave heating

In a recent paper Croquesel et al. (Materials and Design 88 (2015) 98-105) presented a 2.45 GHz monomode cavity furnace allowing direct microwave heating of pure ceramic powder. This cavity was optimized with an impedance tuner so as to maximize electric power to the specimen. It includes an optical dilatometry device that allows following the shrinkage during sintering and a pyrometer-based technique specifically calibrated for reliable measurement of the sample temperature in the microwave furnace environment. With this equipment, direct microwave sintering can be significantly compared with conventional sintering and specific effects of microwaves on the sintering behaviour, if any, can be evidenced. Three nano gamma-alumina powders were used: two non-doped powders seeded with 3.3 and 9.4 % of alpha phase and a 500 ppm MgO doped powder (4% alpha seeded). The sintering behaviours in the 2.45 GHz monomode microwave cavity and in a conventional dilatometer were carefully compared. The microwave experiments were performed in direct as well in hybrid (SiC susceptor) conditions.

Reference conventional and microwave experiments were performed at the same constant heating rate (25°C/min) up to 1550°C. The master sintering curve model was used to extrapolate conventional sintering curves at high heating rates (up to 300°C/min) in order to compare the results with microwave sintering curves. For both undoped powders, the gamma$\rightarrow$alpha phase transformation is shifted by 100°C towards lower values in the case of microwave heating. The density jump associated to the phase transformation, which increases with seeding in conventional sintering, is almost independent of the alpha content in microwave sintering. With a high amount of seeding (9.4%) the final densities and micro-structures with the same thermal cycle are equivalent. Mg doping strongly reduces densification in alpha phase after the transformation in microwave sintering, which is not the case in conventional sintering.

The behaviour of doped and undoped powders is explained by a specific enhancement of interface mechanisms by the field, and the modification of this interaction by the doping elements.