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ADVANCED CERAMIC TECHNOLOGY FOR SPACE APPLICATIONS AT NASA-MSFC

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Introduction

The ceramic processing technology using conventional methods is applied to the making of the state-of-the-art ceramics known as *smart ceramics* or *intelligent ceramics* or *electroceramics*.[1,2] The *sol-gel* and *wet chemical* processing routes are excluded in this investigation considering economic aspect and proportionate benefit of the resulting product. The use of ceramic ingredients in making coatings or devices employing vacuum coating unit is also excluded in this investigation. Based on the present information it is anticipated that the conventional processing methods provide identical performing ceramics when compared to that processed by the chemical routes. This is possible when sintering temperature, heating and cooling ramps, peak temperature (sintering temperature), soak-time (hold-time), etc. are considered as variable parameters. In addition, optional *calcination step* prior to the sintering operation remains as a vital variable parameter. These variable parameters constitute a *sintering profile* to obtain a sintered product. Also it is possible to obtain identical products for more than one sintering profile attributing to the *calcination step* in conjunction with the variables of the sintering profile. Overall, the state-of-the-art ceramic technology is evaluated for potential thermal and electrical insulation coatings, microelectronics and integrated circuits, discrete and integrated devices, etc. applications in the space program.

The ceramic systems are randomly oriented single/poly-phase polycrystalline semiconductors. These systems are based on oxides or non-oxides or some sort of hybrid composites comprising of both. Lightweight ceramic materials are continuously searched for variety space applications as sensors, microelectronic devices and circuits, insulators, coatings, radiation shielding, energy conversion, mechanical and structural support, etc. Utilizing traditional ceramic processing methods followed by sintering in conjunction with the calcining step is emphasized for better performing ceramic body. It is visualized that traditional ceramic processing methods are economic routes for making active stable devices, corrosion preventing coatings, non-degrading insulators and structures, etc. Therefore, smart ceramics imply effective ceramic bodies that are successfully used in the severe or the hostile application field without failure or having increased longevity.

Processing/Fabrication of Ceramics

The ceramic processing technique involves slurry and spray-dried granule preparation for conventional sintering using high temperature kiln. The *microwave sintering* and the *laser sintering* are not included in this investigation. The slurry preparation depends on the raw ingredients as the surface charges of the particles play an important role to constitute *zeta potential*. The zeta potential results from the lumped surface charges contributed by each particle from the dangling bonds. The nature of the charge density dictates the *p*H of the slurry and, thus, related to the zeta potential. In general, high zeta potential indicates *well-dispersed* slurry while low zeta potential indicates *weakly or strongly flocculated* slurry. In addition, agglomeration of the particles is also a severe issue caused by the van der Waals surface forces. Agglomerated powders do not fill the space efficiently. Both flocculation and agglomeration result in voids in the microstructure of the final product.

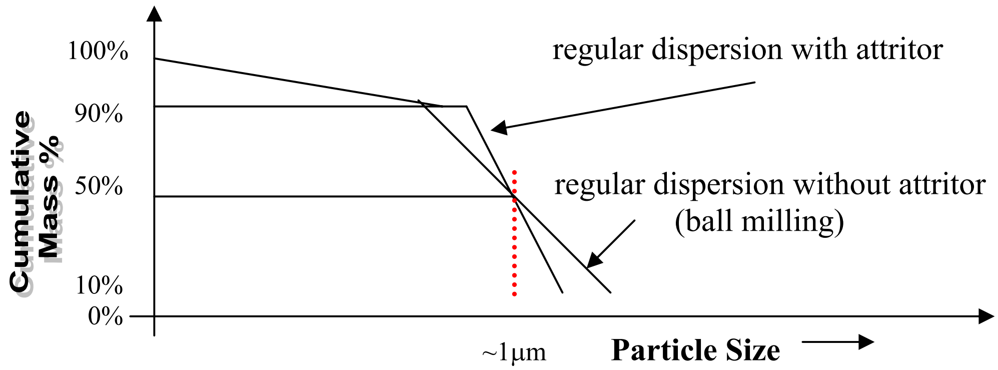

Figure 1: Particle size analysis of the powder.

The *p*H controlled *organic surfactants* are used as the dispersing agent to obtain better dispersion by removing flocculation and agglomeration. The organic binder is also used during the slurry preparation that also plays a role in achieving well-dispersed slurry possessing low viscosity of the non-Newtonian fluid. These organics are removed via the binder-burn-out operation prior to sintering. Again, the uniform particle size distribution plays vital role for the well-dispersed slurry. The particle size is measured at *50 cumulative mass percent* shown in Figure 1. The attritor milling system usually improves the distribution pattern of the particle size via narrowing down the broader distribution obtained by the conventional ball milling. After the slurry is prepared, granular powder is obtained via spray drying. The near loss-less condition for the amount of the granular spray-dried powder is achieved via the adjustment of the *orifice size* and *nozzle air pressure*. The granular powder, containing moisture, is used for pressing into desired geometry.

Effect of Sintering

The sintering operation can be initiated for both *calcined* and *non-calcined* processing routes. When the ingredients are calcined they need to be processed again by controlling the particle size for better dispersion and spray-drying operation. *The calcining operation allows forming single-phase or unified crystal structure* usually below 1000^{o}C. It also allows better-dispersed slurry for certain multi-component ceramic systems. The multi-structure particles cause variation in the charge distribution without calcining operation affecting zeta potential. Thus, the calcining step is an additional operation depending on the type of the ingredients. The calcined phase may be treated as the single-phase like system or single-cation like dopant in the host material.

The sintering profile uses desired heating and cooling ramps with a soak-time (hold-time). *These ramps aid to develop desired electrical, mechanical, and structural characteristics*. Typical sintering profiles are depicted in Figure 2. More than one sintering profile can provide products possessing identical electrical, mechanical, and structural properties.[3,4] However, the degree of homogeneity in the operative electrical paths between the electrodes across the sample may vary. Each polycrystalline material exhibits *trapping effect* that can be minimized by adjusting the sintering profile. Each sintering profile may be replaced by another sintering profile via the manipulation of the heating and cooling curves or soak-time causing identical non-Debye response for the resulting products.

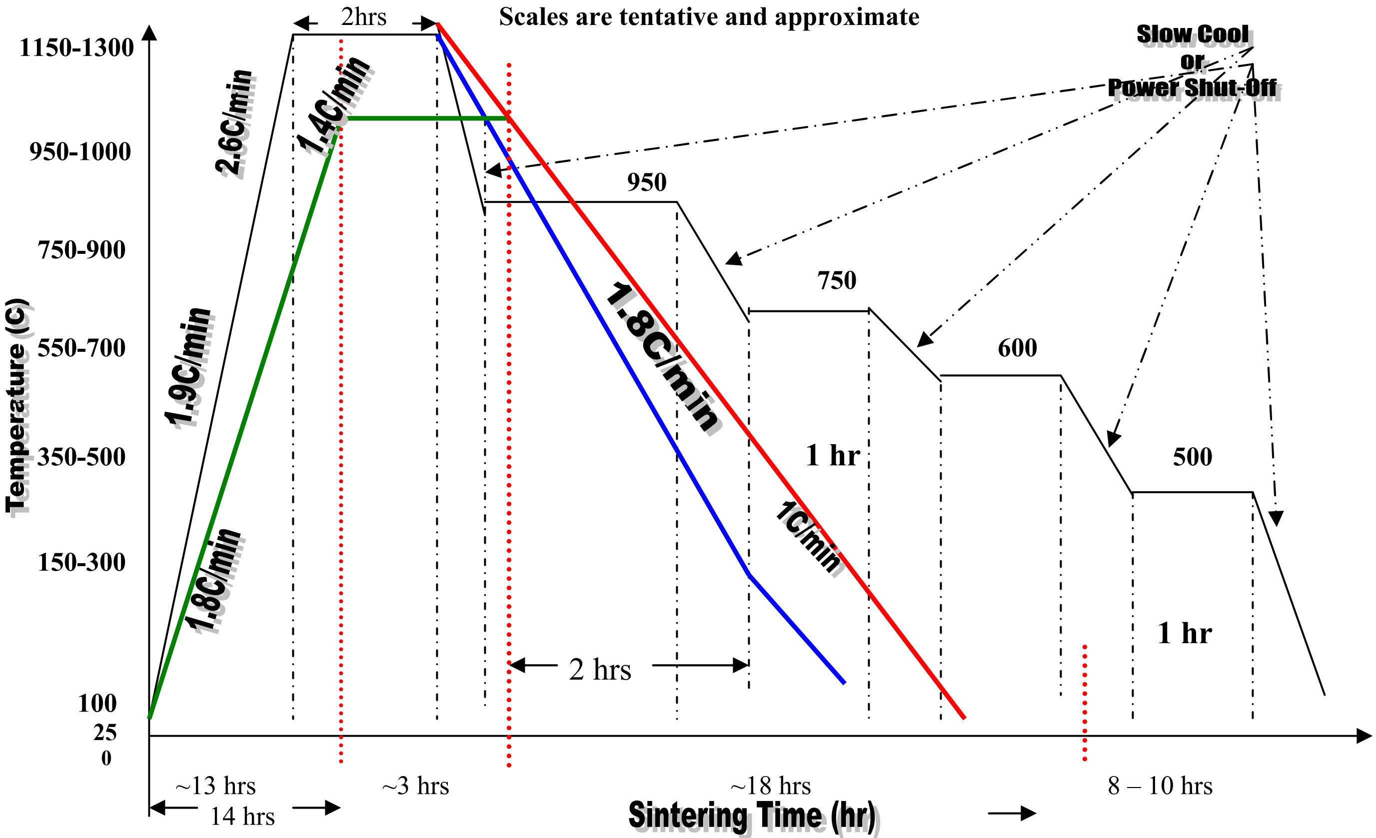

Figure 2: Sintering Profiles.

Sintering operation provides solid-state reaction process. Often causes liquid-phase sintering. Thus, sometimes the resulting shape of the products changes. Also liquid-phase sintering causes better compaction and densification of the sintered body possessing better grain-size. Usually multiple-phase microstructure results from multi-component starting recipe regardless of calcination. The soak-time usually causes the lattice sites adjusted and re-arranged with the doping process of the added cations.[3] The cooling ramp often can be exercised in various forms as the post-heat treatment or annealing of the material.

Figure 3 illustrates the effect of sintering temperature on grain size.[3] It also documents linear increase of grain size with increasing sintering temperature. The dotted and solid curves represent grain size distribution following attritor-milled particles and ball-milled particles, respectively. The distribution of grain size for the attritor-milled particles is narrow regardless of the sintering temperature. Thus, attritor-milled particles provide better microstructures with increased homogeneity via distribution of grain size.

Recommendation and Future Program

A number of oxides and non-oxides have been procured for making ceramic bodies for potential space applications. NASA – MSFC needs to set-up both calcining and sintering operations including sophisticated characterization tools/techniques.[1,4] The competitive state-of-the-art ceramic processing technology will be conducive to the desired goals.

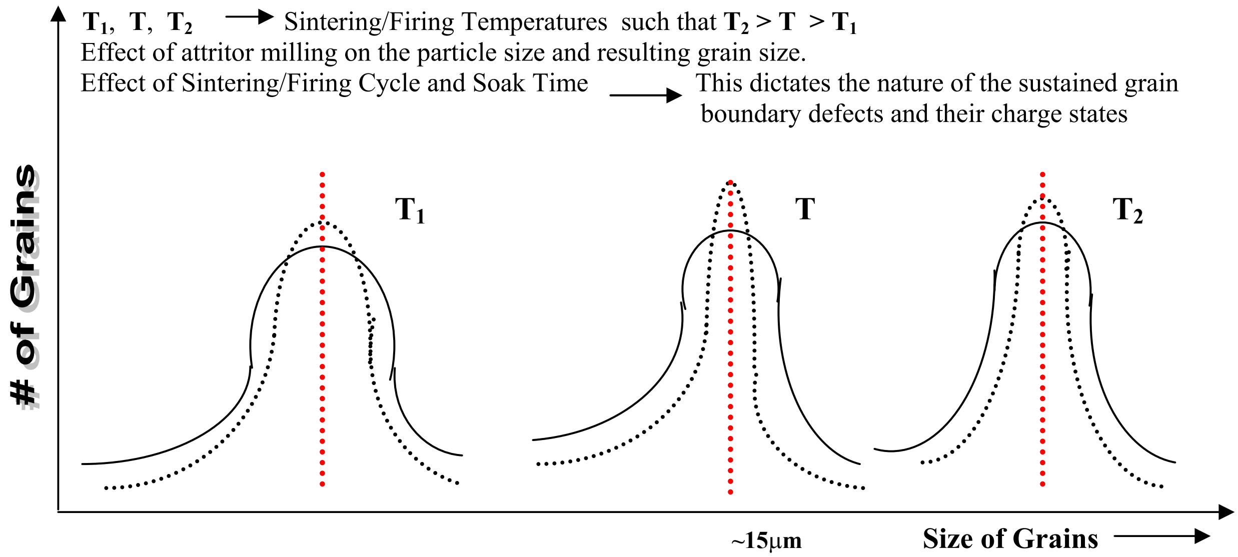

Figure 3: Effect of sintering temperature on grain size.

The existing kilns require precise controllers and real-time monitoring sintering profile (also calcination profile) including leak-less refractory materials. There are several in-house characterization tools such as SEM/EDXS, x-ray diffraction, vacuum coating unit, and etc. available for ceramic evaluation. However, addition of an Impedance Analyzer will be useful to complement information obtained via these characterization tools. Necessary information on the instrumentation such as MIC or Omega or Partlow temperature controllers, and the manufacturers of the kilns such as Harrop or Harper or Lindberg Blue M is provided to the NASA colleagues. In the near future additional Impedance Analyzer(s) may be needed to expand the frequency range data so that a broad window of interpretation becomes easy with better precision.

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