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Thermal shock resistance of refractory composites with Zirconia and Silicon-Carbide inclusions and alumina binder

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Keywords: Thermal shock Particle-reinforcement Composite Refractory Castable Ceramic	With the goal of designing a castable refractory for an aerospace application with optimum resistance to thermal shock, three different particle-reinforced ceramic composites are designed and compared. Different volume fractions of Silicon Carbide (SiC) particles, Zirconia (ZrO ₂) bubbles, and Zirconia solid particles dispersed in an alumina (Al_2O_3) matrix are used in the fabrication of these castables. Destructive and nondestructive testing procedures are implemented to evaluate their thermomechanical properties, both before and after a custom designed thermal shock cycle. After demonstrating the applicability of thermal shock indices, the variation of these indices due to thermal shock is measured experimentally and utilized as a design tool. Multiple micro-scale
	damage mechanisms were observed, all of which are various forms of structural deformation.

1. Introduction

Refractories can be made of various compositions as well as through different production processes, which in turn would lead to a diverse range of properties. Consequently, refractories can be classified in various ways based on each of their major properties [1]. Some important properties of refractories include: resistance to high temperatures and thermal shock, chemical inertness, resistance to mechanical load, resistance to corrosion, erosion and impact [2,3]. From another perspective, refractories can also be classified based on how they are shaped and implemented in the structure. Some refractories come in pre-formed shapes, whereas some others can be shaped in situ and without joints so as to form an integral component [4,5]. This latter group of refractories is called monolithic refractories [4]. One subgroup of monolithic refractories is castables [5]. Castables are composed of refractory grains, which are dispersed in a binding matrix. The castable, depending on the amount of this binding matrix material, can be a selfflowing castable or a vibration castable [6]. After addition of a suitable liquid (e.g. water), the solution is poured into the target location to form the desired refractory shape or structure after solidification due to chemical reaction [4].

Among different properties of interest for castables, thermal shock resistance is arguably the critical one, as castables are frequently subjected to severe thermal loads. In this work we examine the thermal shock resistance of castable refractories made of ZrO_2 and SiC inclusions and hydratable alumina binders. In the remainder of this section, a brief summary of the current state of knowledge in thermal shock resistance of castables is presented, which provides the context for our work described in the subsequent sections.

1.1. Thermal shock in refractories

The presence of a temperature gradient can give rise to thermal stresses in solid materials [7]. However, if this temperature gradient is applied suddenly, it can lead to "thermal shock" [7]. This temperature gradient can be the result of sudden heating, which leads to hot shock, or it can be caused by sudden cooling, which causes cold shock [8]. Thermal shock resistance can be defined as the ability of the material to withstand different forms of failure that may take place during rapid cooling or heating [7,9]. Thermal shock resistance is not an intrinsic property of a material and it is strongly related to the size [10] and shape [11] of the material as well as duration and the method by which thermal gradient is applied [8,9,12]. Nonetheless, some of the properties of the solid that can affect its thermal shock resistance include coefficient of thermal expansion (CTE) [7,8,13,14], thermal conductivity [7,8], tensile strength [7,8], modulus of elasticity [7,8], toughness [8], thermal diffusivity [7] and Poisson's ratio [7,15]. For metals, thermal stresses may cause small plastic deformations; whereas in contrast, due to linear elasticity of ceramic materials, large stresses can be generated in response to thermal shock [16].

In order to analytically predict the resistance of homogeneous ceramics to thermal shock, multiple parameters have been proposed

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[7,17]. Although as previously emphasized thermal shock resistance depends upon various factors, including the shape of the sample, the aforementioned thermal shock parameters are defined in terms of intrinsic properties of the material only. One such thermal shock parameter is R, which is defined as follows [7]:

$$R = \frac{\sigma f(\nu)}{E\alpha} \tag{1}$$

where E, α , and ν represent elastic modulus, CTE, and Poisson's ratio, respectively. Also, parameter σ can either represent tensile strength (σ_t) in the case of cold shock, or represent crushing strength (σ_c) in the case of the hot shock. The definition of the function $f(\nu)$ depends on the state of stress. The value of $f(\nu)$ can be equal to 1 or $(1-\nu)$ or $(1-2\nu)$ for uniaxial, biaxial and triaxial stress, respectively. The parameter *R* describes the maximum allowable temperature gradient that the material can withstand without crack initiation, and it is used for hard thermal shocks where the Biot number is relatively high. Biot number can be defined as [8]:

$$\beta = \frac{bh}{\lambda} \tag{2}$$

where *b* is a thickness, *h* is the coefficient of surface heat transfer and λ is the thermal conductivity. In a similar fashion, the criteria for mild thermal shock, *R*', is defined as follows [7]:

$$R' = \lambda \frac{\sigma f(v)}{E\alpha} \tag{3}$$

The *R*' parameter describes the maximum allowable heat flux, and it is used in cases where the Biot number is relatively low. Together, *R* and *R*' parameters can provide us with a useful description about crack initiation in a ceramic material due to cold or hot thermal shock, both of which can take place either by hard shock or by mild shock regimes. From a different perspective, another category of thermal shock parameters are developed to describe the resistance of the ceramic to crack propagation. One such parameter is *R*'' defined as follows [7,17]:

$$R^{t''} = \frac{E}{\sigma^2 f(\nu)} \tag{4}$$

which from the perspective of strain energy, describes the resistance to spalling. As can be inferred from their corresponding equations, resistance to crack initiation and crack propagation are two qualities of the ceramics that are competing with one another, and depending on the application, one may be preferred to the other [17]. In general, at least two thermal shock parameters (or merit indices) need to be implemented to characterize the response of material to various conditions of thermal shock, and no single one of these parameters would be adequate independently [7]. Fig. 1 qualitatively describes how each pair of the aforementioned indices can evaluate certain aspects of thermal shock response of the material under examination, collectively.

Notice that these parameters, in conjunction with many other thermal shock indices are essentially established to portray the thermal shock resistance of homogeneous ceramics; thus, their validity for describing composite materials such as refractories can be considered as ad-hoc [18]. Also, they are not exploited for characterizing the thermal shock cycle (the temperature profile) applied to the ceramic materials. Moreover, for ceramic composites the crack initiation and crack propagation steps can be more intertwined; thus, implementing both groups of parameters simultaneously for characterizing the thermal shock response seems to be a reasonable approach, as has been done by other researchers [19–21]. With this background, in the present work we start by hypothesizing applicability of the thermal shock parameters for describing thermal shock resistance of our composites, and after proving the hypothesis, implement them as comparative design tools.

1.2. Experiments on thermal shock

In the existing body of experimental research on thermal shock resistance of refractory materials, different quenching techniques have been designed for simulating cold shock. The specimen may be placed in contact with a cold metal rod, or immersed in a quenching medium. For the latter technique, different quenching media have been implemented, such as room-temperature water, boiling water, room temperature air, different types of oils and alcohols, and preheated salt [15,19–32]. Hot shock is also simulated experimentally by means of a flame [33–36], contact with molten metal bath [37] and many other methods [28], but most frequently by using different types of furnaces in the lab [37–40].

Damage assessment of thermal shock can be performed by means of destructive as well as non-destructive testing procedures. Measuring changes in elastic modulus as a result of thermal shock, using either ultrasonic method or resonance method has been one of the most common nondestructive practices for characterizing thermal shock damage [37-42]. Among destructive methods of characterizing thermal shock damage, wedge splitting, three-point-bending, and compression tests are the most common procedures for measuring strength, modulus of rupture (MOR), work of fracture and other properties [12,21,23–25,31]. In addition to the previously mentioned destructive and nondestructive tests, Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), as well as other imaging techniques are frequently practiced to provide insight about microstructure, changes and failures due to thermal shock, and crack deflection mechanisms [19-24,36,43,44], or even implemented as an independent tool for characterizing thermal shock damage by inspecting morphology of cracks, density of crack, etc. [31,45-47].

Thermal shock resistance of refractories may be improved from the perspective of crack initiation, as well as crack propagation [44,48]. To prevent crack initiation, properties of the material need to be optimized to obtain higher thermal shock crack initiation indices [44,48]. As an instance, increasing strength and decreasing elastic modulus would be helpful according to Eq. (1). Ye et al. [49] reported improvement in the strength of the hydratable alumina-bonded castable through the



Fig. 1. Qualitative description of (a) The influence of R and R' on response of materials to hard and mild thermal shocks, and (b) the influence of R and R'' on response to damage initiation and propagation.

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