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Development of the Dynamic Compaction Resistance Sintering (DCRS): A new process for powder consolidation combining electric current and dynamic loading

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ABSTRACT

A homemade powder consolidation process combining high heating rates obtained by resistive sintering and dynamic compaction loading has been developed. The Dynamic Compaction Resistance Sintering (DCRS) device is fully automated and allows a complete regulation of the loading and temperature cycles under controlled atmosphere conditions. The general principle is to carry the electric current for sintering by means of copper Hopkinson bars. Thus, in addition to the conventional application of a constant uniaxial loading (5–100 MPa), the Hopkinson bars offer the possibility of multiple dynamic loadings at velocities up to 30 m/s. The DCRS device was tested at temperatures as high as 1850 °C and for heating rates up to 1000 °C/min. Some of the potentialities of this technique are illustrated through the low-temperature consolidation of copper under repeated dynamic loadings and high-temperature sintering of alumina.

1. Introduction

Powder metallurgy techniques are employed to process a large spectrum of materials from metals to ceramics and their composites. According to Fleck et al. (1992), pressure applied during the sintering cycle helps densification by plastic flow. As a consequence, it helps to remove residual porosity. Among the powder metallurgy processes, Hot Pressing (HP) is effective in applying pressure but does not allow high heating rates to be applied (typically < 20 °C/min), which is not suitable for processing nanomaterials because of microstructural coarsening (Groza, 2007). For a decade, new technologies allowing higher heating rates have emerged. Among them, the Field Assisted Sintering Technology (FAST) – also referred to as "Spark Plasma Sintering" (SPS) – combines pressure with direct heating by high current under low voltages generating a Joule effect within the material. The complete review by Orrù et al. (2009) highlights the FAST effectiveness compared to other processes for a large range of materials, especially for refractory materials such as intermetallics (Ji et al., 2006), tungsten carbide (Grasso et al., 2009) or molybdenum (Ohser-Wiedemann et al., 2010). While the majority of FAST studies have focused on the effects of thermal parameters such as heating rate (Aman et al., 2010), dwell time (Aman et al., 2011) and sintering temperature (Demirskyi et al., 2011), only a small number of them have investigated the influence of pressure in detail (Grasso et al., 2011).

Pressure plays an important role during sintering. Its effect can be generally adjusted through the magnitude of its intensity or by its variation throughout the sintering cycle. According to Anselmi-Tamburini et al. (2004), the application of pressure increases the driving force for sintering and allows the sintering temperature to be lowered. The same conclusion was reported by Munir et al. (2006). Wang et al. (2011) demonstrated that a multistep pressure consolidation procedure could give better results than a single-step one in terms of the optical properties for alumina powder. Indeed, these authors applied a two-step pressure procedure

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during FAST sintering: a low pressure during heating and a higher pressure at the final sintering temperature. The evacuation of gas trapped in the material is facilitated at low temperature while plastic deformation, which facilitates densification, is enhanced at high temperature. This results in a finer grain size and a better homogeneity than with the single-step procedure. The effect of pressure, and the temperature at which it is applied, was also demonstrated by Diouf and Molinari (2012) for metallic powders such as copper. Thus for both ceramic and metallic powders, it is clear that the loading procedure also influences the material properties. Along with its importance during the sintering process itself, Aman et al. (2009) demonstrated that pressure also plays an essential role during the precompaction stage required to prepare the green bodies prior to starting the actual FAST process. This process, which involves particle rearrangement, deformation and possibly fractures, is usually performed under a quasi-static loading mode conditions. However, Aman et al. (2009) showed that these conditions are not necessarily well adapted to all types of powders. Thus, instead of a precompaction in a quasi-static mode, the review of Gourdin (1986) points out that the dynamic compaction of powders may be attractive as it provides higher green strength and a more uniform green density distribution.

Dynamic compaction generally includes a number of techniques, from the laboratory scale to the industrial scale, in which the powder is subjected to an intense shock wave generated either by detonating an explosive (Murakoshi et al., 1993), by the impact of high-velocity projectiles and flyer plates (O'Donnell, 1987) or by using of high-mass hammering tools (Kurita et al., 2005). This stress wave, characterized by a short duration (typically < 1 s), propagates through the material, leading to the compaction and bonding of the powder particles. Thadhani (1988) has demonstrated that the bonding mechanisms in shock compaction involve the rapid and intense concentration of shock energy, preferentially in interparticle regions, resulting in extensive plastic deformation. Although the Split Hopkinson Pressure Bar (SHPB) is usually dedicated to the characterization of solid materials at high strain rates, Häggblad et al. (2005) with metallic powder and Trecant et al. (1995) with ceramic powder have already tried to use it for cold powder compaction. Additionally, while using a High-Velocity Compaction (HVC) device, Eriksson et al. (2003) observed a cumulative effect of the energy of impact on the green density when performing several impacts. Thus, HVC has been used to form ceramic compacts (Souriou et al., 2009) as well as Titanium (Yan et al., 2011) and Ferrous (Wang et al., 2009) green-bodies having better homogeneity than their counterparts obtained by conventional uniaxial pressing. While the HVC technique has also been used recently to sinter, by self heating and local melting, semi-crystalline polymer powders (Jauffrès et al., 2009), it is only recently that Tang et al. (2010) used gravitational potential energy to perform one strike of HVC in a forging mold heated by coils and, thus, obtain samples having a higher density. However, to the authors' knowledge, the effect of multiple dynamic loadings coupled with the effectiveness of the FAST technology has never been tested for powder sintering. For example, the heating SHPB (6000 K/s) developed by Basak et al. (2004) had another purpose, i.e. the dynamic compression testing of a bulk material at a high temperature. Interestingly, while shock waves are generated in the material using the above mentioned techniques like explosion, HVC or even by impact using flyer plates (Yu and Meyers, 1991) – with a strong reflecting wave which may also promote the formation of voids and cracks –, the energy imparted to the material by the SHPB is not high enough to create such a strong effect. Also, another advantage of using the Hopkinson bars to heat up the sample is that, contrary to HVC and flyer plates, fast thermal cycles can be apply. Thus, the goal of our research was to develop a new apparatus combining the advantages of the FAST heating coupled with one to multiple dynamic loadings by Hopkinson bars in order to improve the sintering of ceramic and metallic powders.

This paper describes a new process – called DCRS for Dynamic Compaction Resistance Sintering – which offers a combination of high heating rates with the dynamic compaction of powder materials and illustrates its ability to improve the sintering process. The SHPB was chosen as a dynamic compaction device because it is possible to associate it with a FAST heating system. The description of the process and of its constitutive elements are presented in the first part of this paper, depicting the specific design considerations taken into account for the temperature generation and control (Section 2.1) as well as the application of the different loadings (Section 2.2). In the second part of this paper, the accuracy and potential of this new technique is detailed. A thermal validation consisting of ensuring the correct temperature regulation as well as its reproducibility on both an electrically conductive metallic (copper) powder and an electrically insulating ceramic (alumina) powder is presented. The effect of the dynamic impact on the material consolidation at the end of a thermal cycle is then investigated for the copper powder.

2. Specific design considerations

Fig. 1 depicts the schematic of the DCRS device. It basically consists in adapting an electrically resistive heating device on top of an SHPB. It has been designed for powder sintering in order to provide with the following capabilities:

- working temperatures as high as 2000 °C,
- heating rates ranging from 1 °C/min to 1000 °C/min,
- controlled atmosphere (vacuum or gas),
- uniaxial pressure ranging from 5 to 100 MPa,
- dynamic loading at velocities up to 30 m/s,
- sample diameter of 12 mm.

The rapid thermal parameters are within the ranges of those which can be obtained for the conventional FAST devices, but the DC current is applied through the copper-based Hopkinson bars which are also used for the dynamic loading. As illustrated in Fig. 1, the main features are the input and output bars associated with a pneumatic gas gun used to launch a striker. The dynamic loading is generated by the impact of the striker against the input bar and creates an incident uniaxial elastic wave that propagates toward the material. The major addition to the conventional FAST technology is the possibility of dynamic compaction loading cycles provided by a striker which impacts the consolidating powder at a speed ranging from 5 to 30 m/s. As for the conventional SHPB device, the amplitude and period of the longitudinal wave generated by the striker are determined both by the speed and length of the striker. Its speed can be measured by means of photodiodes located at the airgun output.

2.1. Temperature generation and control

The DCRS, as most FAST devices, uses the Joule effect in order to heat the powder material. As illustrated in Fig. 1, a DC power supply (50 V, 3000 A) providing the Joule heating is connected to the copper bars of the SHPB and the powder is consolidated within a graphite tool (die and punches made of graphite grade 2333, graphite sheets made of Papyex, *Mersen*). Graphite was chosen because of its very low coefficient of thermal expansion and its outstanding temperature resistance (up to 2200 °C). Indeed, according to the manufacturer (Tanso, 2013), the mechanical strength of the graphite grade used here increases with the operating temperature. The temperature of the material is monitored through a blind hole drilled in the graphite matrix using both thermocouples and