



# Production of nickel matrix composites reinforced with carbide particles by granulation of fine powders and mechanical pressing



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## ABSTRACT

Plain nickel and nickel composites containing either TaC or NbC powders were granulated and sintered after conventional mechanical pressing. The granulation parameters were studied using plain nickel powder to evaluate a binder system consisting of paraffin diluted in hexane, but also the suitable size range of the granules obtained. Carbide powders were then wet mixed with nickel and the powder mixtures were granulated using a drum. Flow rate tests were performed and the granules were pressed into pellets, which were dewaxed and sintered. SEM images of the microstructure of the sintered materials were obtained. Green and sintered densities along with Brinell hardness were also evaluated. The results revealed that the concentration of 1.5 wt% of paraffin and granules sieved in the range between 500 and 90  $\mu\text{m}$  contributed towards the development of dense and uniform microstructures of the sintered composites. Finally, the addition of carbides homogeneously increased hardness to nearly double that of plain nickel.

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## 1. Introduction

Powder granulation has been investigated for almost 70 years. Studies report the use of a variety of methods and equipment, including drums [1–2] applied to different materials, ranging from minerals to metallic powders of industrial interest [3]. Granulation has been extensively used to improve the flow rate of powders aiming at improved die feeding for mechanical pressing. High flow rates avoid defects such as mass heterogeneities in the green parts. Consequently, granulation yields homogenous density of pressed parts which results in dense and homogenous sintered products [4] characterized by superior physical characteristics and mechanical properties [5]. Granulation is especially recommended for fine powders, which normally exhibit low flow rates [6,7] and are difficult to handle and press using simple mechanical equipment. Defects originated from inappropriate die filling cannot be easily removed by further processing. Therefore, granulation is an important step to assure proper technological performance of sintered parts.

Powder granules are produced from a suspension of solid particles that agglomerate in a liquid phase [8–10]. The surface of the particles

and the binding liquid must be of identical polarity [11–12]. The liquid binds the particles together by a combination of capillary pressure, surface tension, and viscous forces to a point where permanent solid bridges are formed after evaporation of the solvent [13–14]. Two attractive interparticle forces take place. Electrostatic forces are responsible for the initial agglomeration, however, they do not significantly contribute to the final strength of the granules, defined by van der Waals attraction, whose magnitude is usually four times higher [15,16]. Powder granulation is, in fact, an intricate process involving a number of simultaneous physical phenomena: i) wetting, nucleation and binder distribution; ii) consolidation and growth and iii) attrition and breakage [17–18]. A minimum amount of binder is determined by both powder and binder characteristics. Other important aspects of the process include binder distribution in the powder volume and time required for binder spreading [14]. The liquid–powder mixture is stirred to promote dispersion and growth of the granules, which are formed by the collision and adhesion of primary particles in discrete granules and/or growth around a core where the particles collide and aggregate to form layers.

Granulation can be performed in a drum. This method is especially useful to process small powder batches and to reduce production costs [19]. A binder and its solvent are added to the powder mixture and the batch is rotated in the drum. As the solvent evaporates, the particles coalesce in granules. The spreading efficiency of the binder solution is the main controlling feature of the process; however, binder selection for a particular powder system is quite often empirical [20].

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Final granule size distribution mainly depends on processing parameters such as residence time of particles in the drum and rotation speed [21].

Metal matrix composites (MMCs) can be manufactured by powder metallurgy (PM). Sintered parts have gained growing attention and achieved significant market expansion. An increasing number of sintered metallic components with improved performance and manufactured at low cost [22] has been successfully produced. PM involves a small number of energy efficient processing steps and results in near net shape parts with a wide variety of compositions and complex geometric shapes [23]. The production of MMCs with dispersed ceramic particles can be easily achieved by powder metallurgy [24]. The metallic matrices provide plasticity, whereas the dispersed ceramic particles adjust several target properties, such as hardness, strength and wear resistance. In addition, the porosity and pore size distribution of the sintered parts can be adjusted to result in either dense components or parts with porosity gradient, potentially expanding the number of applications for these materials.

Nickel is of great interest for its vast use in engineering applications that require some of its unique properties such as magnetism, strength, corrosion and wear resistance at high temperatures. In the latter case, improved properties can be achieved by adding dispersed hard particles such as TaC and/or NbC into Ni powder and sintering to nearly full density [25]. The main problem in the manufacture of composite parts with fine reinforcing ceramic particles is their distribution in the microstructure of the sintered component [26]. In order to obtain homogeneous distributions, and therefore, little property variation along the microstructure, fine powders are preferable; therefore, granulation of the powder feedstock becomes important. In this scenario, the main goal of this work was the study the granulation of Ni-TaC and Ni-NbC powders to produce nickel-based sintered metal matrix composites by conventional powder metallurgy.

## 2. Materials and methods

### 2.1. Materials

The materials used were carbonyl nickel powder ( $D_{50} = 6.00 \mu\text{m}$  and  $\rho_{\text{th}} = 8.9 \text{ g/cm}^3$ ) provided by Epson Atmix Corporation (Japan), TaC ( $D_{50} = 2.18 \mu\text{m}$  and  $\rho_{\text{th}} = 13.9 \text{ g/cm}^3$ ) and NbC powders ( $D_{50} = 2.26 \mu\text{m}$  and  $\rho_{\text{th}} = 6.3 \text{ g/cm}^3$ ) supplied by H. C. Starck (Germany). SEM micrographs of the starting powders can be seen in Fig. 1. Commercially available paraffin lentils and hexane solvent were used to granulate the feedstocks.

### 2.2. Methods

The granulation parameters and the appropriate particle size range of the granules were initially studied. Granulation of plain Ni was performed using different contents of paraffin, i.e., 0.5, 1.0 and 1.5 wt%, diluted in hexane in ultrasound bath for 30 min in a sealed container. Subsequently, 100 g of powder was added to the solution, which was then transferred to a drum. The granulated powders were separated using 500–90  $\mu\text{m}$  and 850–180  $\mu\text{m}$  sieves. The granules were then pressed, sintered, and the density, microstructure and Brinell hardness of the resulting samples were characterized. After defining the adequate contents of paraffin and the adequate granule size distribution, different concentrations of TaC or NbC powders (Table 1) were wet mixed with Ni using ethanol and 200 g of steel spheres (diameter of 0.3 cm). Powder batches of 150 g were mixed during 1 h. After drying in an oven at  $\sim 70 \text{ }^\circ\text{C}$  for about 2 h, the powders were granulated using 1.5 wt% paraffin, and the granules were classified using 500 to 90  $\mu\text{m}$  sieves.

Flow rate tests were carried out using the Hall funnel with orifice diameter of 0.20 in., according to ASTM B213-13 [27]. The granulated powders were pressed under 600 MPa in double-acting mode. To minimize the friction between die and green compacts, amide wax soaked

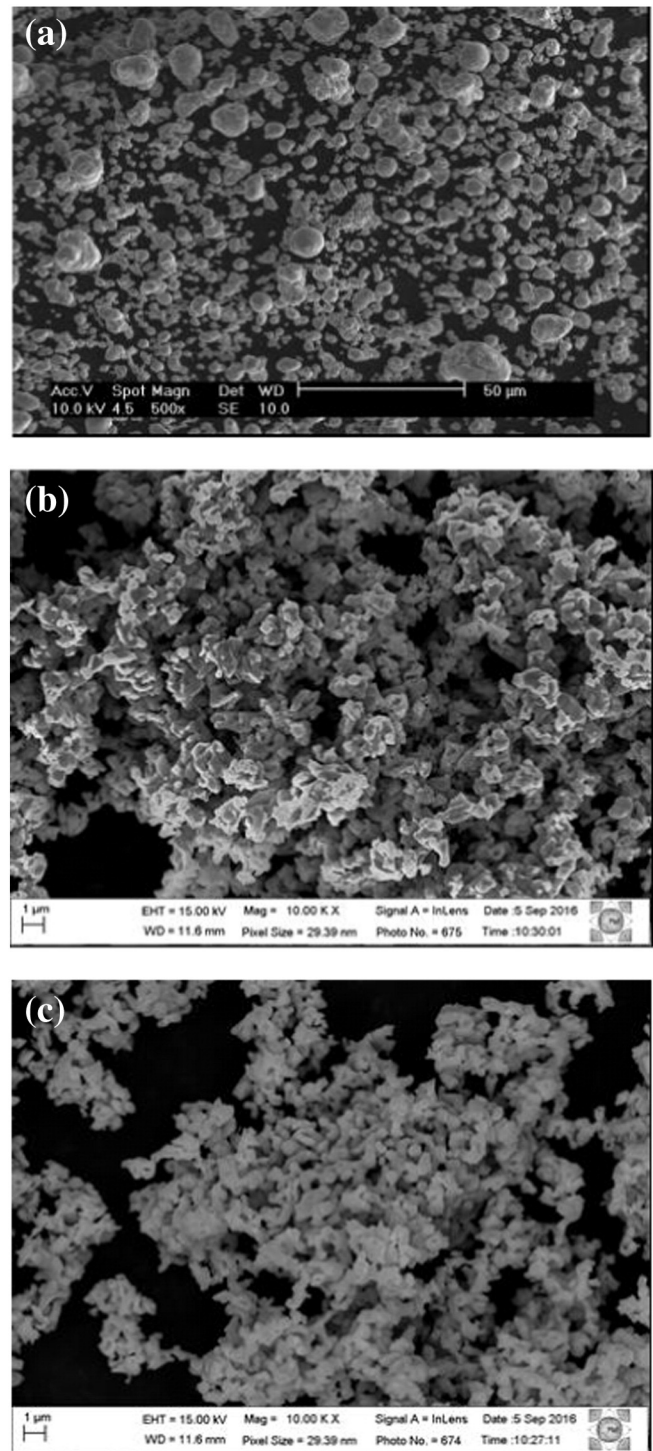


Fig. 1. SEM images of (a) Ni, (b) TaC and (c) NbC powders.

Table 1  
Composite powder compositions and identification.

	N	NT5	NT10	NT15	NN5	NN10	NN15	NTN2.5	NTN5	NTN7.5
TaC										
(wt%)	0	5	10	15	0	0	0	2.5	5	7.5
(vol%)	0	3	6	10	0	0	0	2	3	5
NbC										
(wt%)	0	0	0	0	5	10	15	2.5	5	7.5
(vol%)	0	0	0	0	6	11	17	3	6	9

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