Powder Technology 237 (2013) 221–227

Contents lists available at SciVerse ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

High energy ball milling (HEBM) of high volume fraction hard-phase composite powder: Production and characterisation of Al_4C_3–27 vol.%NiCrAlY composite powder

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ARTICLE INFO

Article history:
Received 27 July 2012
Received in revised form 11 December 2012
Accepted 16 January 2013
Available online 23 January 2013

Keywords:
High energy ball milling
Al_4C_3
NiCrAlY
Mechanical alloying
Composite powder

ABSTRACT

This work characterises the microstructural development of an Al_4C_3 based composite powder containing 27 vol.%NiCrAlY by high energy ball milling for up to 35 h in air. The observed mechanism of microstructural development was distinctly different to the traditional composite formation mechanism in which hard particles are folded into a ductile metal binder. During milling the isolated NiCrAlY particles were surrounded by a thick dense layer of Al_4C_3 particles, preventing cold welding of the ductile phase. With continued milling the consolidated carbide particles acted as the effective binder through which the NiCrAlY particles were dispersed. It was only after 15–20 h of milling that the NiCrAlY phase became distributed sufficiently to act as a binder in the traditional sense. The microstructural and compositional development of the composite powder as a function of milling time is discussed through analysis by scanning electron microscopy, X-ray diffraction and energy dispersive X-ray spectroscopy.

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1. Introduction

Carbide based composites based on chromium carbide are routinely applied by thermal spraying to produce wear resistant coatings. A key factor in optimising the spray parameters for such coatings is the minimisation of carbide dissolution during spraying in order to retain the same carbide concentration, morphology and distribution in the coating as was in the staring powder. Carbide dissolution is considered to degrade the coating quality as it reduces the concentration of the hard, wear resistant phase, while at the same time reducing the toughness of the metallic binder through saturation of dissolved carbide elements. Heat treatment of carbide dissolution zones can lead to the formation of a fine grained interpenetrating network of the carbide phase within the metallic phase [1]. This carbide morphology is distinctly different from that in conventional carbide composites where individual carbide particles are dispersed within a continuous metallic binder. By controlling the amount of the carbide phase, the degree of carbide dissolution and the heat treatment process it should be possible to control the development of this interpenetrating carbide microstructure and thereby tailor the properties of the overall composite.

This concept is currently being explored using an Al_4C_3–NiCrAlY composite system. The Ni–25Cr–6Al–0.4Y (wt.%) binder is a well known super alloy composition used in high temperature applications. The Al_4C_3 hard phase was selected on the basis that it will form a protective Al_2O_3 oxide at high temperature. To explore this concept of microstructure development by thermal spraying the composite must be in the form of a powder of composite particles. In order to generate a high degree of carbide dissolution during spraying the carbide phase within the particles should be as small as possible and finely distributed within the metallic binder. The volume fraction of the carbide phase in the composite powder should be approximately 70–75 vol.% in order to form the same continuous interpenetrating network with heat treatment as shown in previous works [1]. This work details the development of this Al_4C_3–NiCrAlY composite powder through the use of high energy ball milling (HEBM).

HEBM utilises high frequency, high energy impacts from milling balls to repeatedly forge powder particles together [2,3]. In mixtures of ductile components the particles are initially flattened into "pancake-like" morphologies by the forging action of the balls [3]. Cold welding of the flattened particles occurs to form a classical layered or lamellar microstructure. With continued milling the two components become homogeneously mixed to the point where mechanical alloying occurs. In composite systems incorporating a ductile and a brittle component the mechanism is slightly different. The ductile component becomes flattened by the milling action, while the brittle component undergoes size reduction by fragmentation [3]. With continued milling the fine hard phase particles become trapped between the ductile flattened flakes and "folded" into the softer material as it undergoes cold welding [3–5]. This also forms particles with a layered microstructure in which the thin layers of hard particles are retained within thicker lamellae of the ductile binder material. Continued milling refines the composite particle microstructure such that the hard phase particles become homogeneously distributed throughout the ductile binder phase. Alloying

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0032-5910/$ – see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.powtec.2013.01.028

of the binder and hard phase can occur depending on the relative solubility's of the component elements [3].

This technique of composite powder production was initially developed for the manufacture of oxide dispersion strengthened Ni alloys [6]. Since this time it has been widely exploited to improve the mechanical properties of low density metals like aluminium through particulate reinforcement. The concentration of reinforcement in such composites is typically less than 50 vol.%. Few works have investigated the use of HEBM to produce composites with higher hard phase concentrations [5,7–15]. In low hard phase content composites the hard phase particles are folded into the continuous ductile binder. As the volume fraction of the ductile phase is reduced below that of the hard phase this mechanism must change to compensate for the higher hard phase content. To date, however, this mechanism has not been addressed in detail. The aim of this work was to characterise the change in composition and microstructure of an Al_4C_3–27 vol.%NiCrAlY composite powder with milling time to highlight the mechanism of composite formation.

2. Experimental procedure

Aluminium carbide (Al_4C_3) powder (99% purity, nominal size <45 μm) (Sigma Aldrich) and gas atomised Ni–25Cr–6Al–0.4Y powder (NiCrAlY) (nominal size 45–75 μm) (AMDRY 963, Sulzer Metco) were mixed (49.5 g Al_4C_3, 63.1 g NiCrAlY) to give a starting powder composition of Al_4C_3–27 vol.%NiCrAlY. High energy ball milling was performed in a water cooled attritor ball mill (Attritor HD-01) (Union Process) in air using 6.35 mm chromium steel balls with a rotation speed of 600 rpm. The ball to powder mass ratio was 17.7:1. No process control agents were used. Samples were removed after 2, 4, 6, 8, 9, 15, 20, 25, 30 and 35 h of milling. In order to minimise the disturbance of the milling environment, samples were taken from the top of the bed of milling balls using a spoon to remove several balls and the smallest sample possible for characterisation. The sample size was kept to a minimum so as not to significantly influence the ball to powder ratio. The as-supplied and milled powders were analysed by Scanning Electron Microscopy (SEM) (FEI Quanta 200F, USA). Cross sectional images taken using backscattered electron imaging (BSE) were analysed using the software package Image J [16] to determine mean particle sizes and aspect ratios. The elemental composition of the particle cross sections was measured by Energy Dispersive X-ray Spectroscopy (EDS) (EDAX, USA) for the elements heavier than sodium. Based on the SEM analysis an additional trial was run under the same milling conditions to produce powder samples for X-ray diffraction (XRD) (Bruker D8 Advance, Cu source) (Bruker AXS, Germany). Samples were analysed after 6, 15, 20 and 35 h of milling and then returned to the mill to maintain the ball to powder ratio. Three scans were run for each sample — a wide scan ($2\theta = 24–76°$, step size 0.02°), a narrow scan over the main Ni peak ($2\theta = 41–47°$, step size 0.01°) and a narrow scan over a prominent Al_4C_3 peak ($2\theta = 39.1–41.4°$, step size 0.01°). The narrow scans were used to measure the shift in peak position with mechanical alloying and to determine the reduction in grains size [17]. The influence of Cu Kα2 X-rays was removed from the diffraction patterns and the data smoothed using a Savitzky–Golay smoothing filter within the Bruker Diffrac^{Plus} EVA software.

3. Results and discussion

3.1. Starting material characterization

The Al_4C_3 powder morphology ranged from large particles formed from the regular stacking of plate-like grains, through to agglomerates of sub-micron particles, Fig. 1. The lamellar morphology was reflected in the XRD pattern which showed a preferred orientation of several peaks relative to the reference pattern (JCPDS 79-1736), Fig. 2. Peaks of unreacted aluminium left over from the powder production process [18] were also observed.

SEM images of the spherical gas atomized NiCrAlY powder showed that the particles were significantly larger than the carbide grains, Fig. 1. The XRD pattern matched that of the Ni reference (JCPDS 04-0850) in terms of the number of reflections observed and their relative intensity, but was shifted to a higher d-spacing due to alloying effects, Fig. 2.

3.2. Composite powder characterization – microstructure development

The plate-like carbide grains were rapidly broken down in the first 2 h of milling to form equiaxed particles of <5 μm diameter, Fig. 3a. The NiCrAlY particles, which appear as the much brighter particles in Fig. 3a due to their higher atomic mass in BSE mode SEM, were flattened into pancake-like morphologies. The changes in cross sectional area and aspect ratio of the NiCrAlY particles determined using ImageJ analysis of the SEM images from Fig. 3 are shown in Fig. 4. Milling leads to the combination of these compounds into composite particles. The morphology of these particles was distinctly different to that predicted from mechanisms [3–5] proposed from previous studies of ductile phase-brittle phase composite formation during HEBM based on lower hard phase content composites [3,4,6–8,14]. In contrast to all previous models the NiCrAlY particles underwent deformation and size reduction independently of each other. Each NiCrAlY particle was surrounded by a layer of consolidated carbide particles. This layer prevented any interaction or cold welding between the particles of NiCrAlY material, Fig. 3a. Unlike the mechanism discussed in [4,5] the hard carbide particles were not simply forged into the outer layer of the softer alloy binder to form a thin composite periphery. Instead the carbide particles formed a thick, dense consolidated band around each NiCrAlY particle. While the peripheries of the NiCrAlY particles in Fig. 3a were highly distorted and indented by the carbide particles there was limited inclusion of the carbide particles into the NiCrAlY

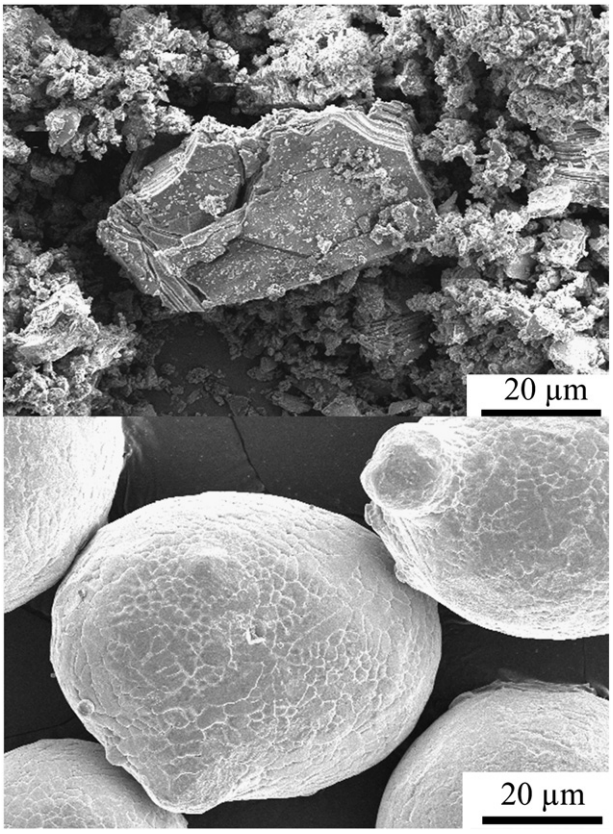

Fig. 1. Topographical images of the as-supplied Al_4C_3 powder (top) and the NiCrAlY powder (bottom).

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