Dynamic compaction of Ni and Al micron powder blends in cylindrical recovery scheme

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Abstract. The experiments on explosive compaction of Ni and Al micron powders mixtures in cylindrical recovery assemblies are reported. Synthesized NiAl compacts were characterized by optical microscopy and x-ray diffraction. The found porosity of the compacts was decreasing with dynamic pressure growth. The geometry of dendrite NiAl grains was associated with the dynamics of pressure waves inside the assembly.

1. Introduction
Nickel monoaluminide NiAl is regarded as a promising structural material, which could be use at temperatures exceeding the nickel superalloys melting points. Now we report on our attempts to prepare NiAl compacts from the Ni and Al micron powders blends via shock-assisted synthesis at high dynamic pressures.

2. Materials
Starting Al powder consisted of rounded granules 1–10 µm in size covered with thin and dense Al₂O₃ coating, while the Ni powder consisted of fluffy conglomerates with a developed surface (2–6 µm in size) formed by sub-micron particles. 0.7Ni + 0.3Al (by wt) powder mixtures were intermixed in a ball mill (mill/ball ratio 5:1, 76 rpm) for 1 hour. The theoretical density (ρₘₐₓ) of this mixture is 5.26 g/cm³. Then the powders were rammed down into stainless steel cylindrical preservation assemblies 801, 802 and 803 (figure 1) to the density ρₚₜ/ρₘₐₓ = 0.55–0.69. Accordingly, porosity ρₚₜ of the rammed powder mixture was 1 − ρₚₜ/ρₘₐₓ = 0.31–0.45.

3. Conditions
The recovery assemblies had an outer diameter of 14 mm (801 and 802) or 10 mm (803) (figure 1). The bottom of the assembly 801 was made in the form of a sharp cone to reduce the pressure. The inner diameter of assemblies 801 and 802 was 8 mm while that of assembly 803.5 mm. All assemblies were 30 mm long. Compaction by detonation products was performed [1, 2] in the explosion scheme represented in figure 2. Sand damper 4 around assembly 802 (figure 2)
Figure 1. Overall view of the recovery assemblies.

Figure 2. Schemes of explosion assemblies 801, 802, 802a, and 803: 1—electrical detonator; 2—HE charge; 3—steel plate; 4—bulk density sand damper.

was used to ensure a longer sample dwelling under pressure. Massive disk 3 at the bottom of assemblies 802 and 803 ensured formation of reflected shock wave and hence some additional pressure increase.

As a rule, 160–300 g HE charges were 57 mm in diameter and 70–100 mm long. The measured detonation velocity (D) had a value of 6.4 km/s. For assembly 802a, the detonation velocity (D) had a value of 7.2 km/s. The velocity of reflected shock wave was estimated as 0.7D [3]. The measured velocity for expansion of detonation products was 2.5 km/s. The estimated radial velocity of unloading wave in detonation products was within the range 0.75–1.2 km/s.

As is seen in figure 3, the amplitude of pressure attained a value of up to 25 GPa at a duration up to 30 µs and more. The primary pressure pulse $P_{DW} = 11$ GPa is of chemical origin and depended on HE density. Behind the detonation front first dropped down to 3.5 GPa and then grew up due to the interaction between the axial reflected waves with $P_{DW} = 25$ GPa and the waves of axial and radial unloading (WuL). In assembly 802a, the acting pressure was higher by a factor of 1.4 due to the use of stronger HE. Recovered samples of compacted were 30 mm long and 5–8 mm in diameter.

4. Results
According to x-ray diffraction data (diffractometer Ultima IV), explosive compaction of overly stoichiometric mixtures (Ni + 48 at. % Al) yielded the NiAl intermetallic. Meanwhile, products 801 and 802 were found to contain Ni ($\leq 2$ % vol.). Product 803 contained also $\text{Ni}_5\text{Al}_3$.
Figure 3. Qualitative pressure on surface profiles for assemblies 801, 802, 802a, and 803: DW is acronym for detonation wave; RSW, for reflected shock wave; and WuL, for wave of unloading.

Figure 4. Optical micrographs of the materials formed near the assembly wall at the mid length.

(~ 5% vol.) but no nickel. This can be associated with a smaller diameter of assembly 803 (stronger external action).

Figure 4 presents the optical micrographs of the materials formed near the assembly wall at the sample middle. The images clearly show the presence of residual porosity. In material 801 the pores exhibit a regular nearly spherical shape with a mean size of 6–13 µm and also some micro porosity (< 1 µm). In material 802 the pores are also rounded at a mean size of 2–6 µm along with inclusions of large (100–200 µm) pores and cracks. In material 803, the uniformly distributed small pores have a size of 0.5–3.0 µm.

It can be assumed that the explosive compaction of NiAl blends proceeded at least in two stages [4]. First, shock-induced SHS reaction initiated near the assembly wall yields dendrite-like intermetallic grains oriented normally to the wall (figure 5a). At the rear end, the dendrite grains are oriented along the sample axis (figure 5b). Near the sample axis, the uniaxial grains of irregular shape were formed (figure 5c). Second, shock waves and subsequent unloading give rise to shear deformation of material leading to formation transverse ruptures (figure 5d) [5].

Figure 6 shows the distribution of porosity comp at the compact surface (solid line) and its center (dashed line) along the direction of wave propagation as determined by using Tescan SEM VEGA-9 TS software. In these results, a contribution from large pores and cracks was excluded as belonging to harmful action of rarefaction waves. The above \( \varepsilon_{\text{comp}}(l) \) functions were found inversely proportional to \( P \) and \( t \): \( \varepsilon^{-1} \sim \int P(t)dt \). In other words, the larger \( P \), the smaller \( \varepsilon \). Accordingly, the behavior of comp for material 803 (figure 6) can be associated with a sharp drop in \( P \) at the middle of assembly due to interaction between the shock and rarefaction waves, which was confirmed by our gas-dynamic estimates. The data of figure 6 were used to estimate the density/porosity of compacts: \( \rho_{\text{comp}} = (1 - \varepsilon_{\text{comp}})\rho_{\text{NiAl}} = 4.5-5.8 \text{ g/cm}^3 \) and \( \varepsilon_{\text{rel}} = (1 - \rho_{\text{comp}}/\rho_{\text{NiAl}})/(1 - \rho_{\text{pr}}/\rho_{\text{max}}) = 0.07-0.39 \).
Figure 5. Micrographs of NiAl taken at different sample sections: a—periphery at the front end of compact, b—periphery at the rear end, c—sample axis, and d—end of compact.

Figure 6. Distribution of compact porosity $\varepsilon_{\text{comp}}$ at the sample surface (solid line) and the sample center (dashed line) along the direction of wave propagation.

5. The further plans
We are planning to carry out similar experiments with Ni-Al nanopowders and with mixtures of Ni-Al nano- and micro-sized powders.

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