

The impact of the operating mode of a planetary mill and gas atmosphere on mechanical alloying of powders in the Ni – Cu system

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Abstract: This study experimentally investigates the formation dynamics of a Ni–Cu solid solution during high-energy ball milling (HEBM) of a Ni and Cu powder mixture in planetary mills. The influence of processing conditions and gas atmosphere composition is examined. It is shown that the formation of a solid solution critically depends on the milling speed: at 300 rpm, no mutual dissolution of metals was observed, whereas at 694 rpm, a solid solution was formed within a relatively short time – no more than 30 minutes. In an oxygen-free atmosphere, intense cold welding of particles occurred, forming large (up to 2–3 mm) disk-shaped and oval flakes, which later became rounded and acquired a spherical shape. In an air atmosphere, cold welding was slowed down, and the metals became less ductile due to oxygen absorption. HEBM in air resulted in the powder consisting of smaller particles with rounded and flake-like shapes (300–400 μm along the surface of flakes, less than 100 μm across). The most homogeneous solid solution structure was formed in an air atmosphere. Thus, the optimal conditions for the mechanosynthesis of metallic solid solutions are processing in a planetary mill at 500–700 rpm in an air atmosphere. The findings of this study can be used to optimize the mechanical synthesis of metallic solid solutions, including Cantor high-entropy alloys with a face-centered cubic structure.

Keywords: high-energy ball milling; planetary mill; microstructure; phase composition; mill mode; gas atmospheres.

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Влияние режима работы планетарной мельницы и газовой атмосферы на механическое сплавление порошков в системе Ni – Cu

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Аннотация: В работе экспериментально исследована динамика формирования твердого раствора Ni–Cu в процессе высокоэнергетической механической обработки (ВЭМО) смеси порошков Ni и Cu в планетарной мельнице, в зависимости от режима обработки и состава газовой атмосферы. Показано, что образование твердого раствора критически зависит от скорости планетарной мельницы: при скорости 300 об/мин взаимного растворения металлов не наблюдается, а при 694 об/мин твердый раствор образуется за относительно короткое время, не более 30 мин. В бескислородной атмосфере происходит интенсивная холодная сварка частиц, образуются крупные (до 2...3 мм) дискообразные и овальные пластинки, которые затем окатываются и приобретают шарообразную форму. В атмосфере воздуха холодная сварка замедлена, а сами металлы становятся менее пластичными из-за поглощения кислорода. Это приводит к тому, что после ВЭМО в воздухе порошок состоит из более мелких частиц округлой и пластинчатой формы (до 300...400 мкм вдоль поверхности пластин, менее 100 мкм поперек). Наиболее однородная структура твердого раствора формируется при обработке в атмосфере воздуха. Таким образом, оптимальным режимом механосинтеза металлических твердых растворов следует признать обработку в планетарной мельнице со скоростью 500...700 об/мин в воздушной атмосфере. Сделанные выводы могут быть использованы для оптимизации механического синтеза металлических твердых растворов, в том числе высокоэнтропийных сплавов Кантора с гранцентрированной кубической структурой.

Ключевые слова: высокоэнергетическая механическая обработка; планетарная мельница; микроструктура; фазовый состав; режим мельницы; газовые атмосферы.

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Introduction

Planetary ball mills are widely used for grinding, alloying, and mechanochemical synthesis of various powder materials [1–4]. High-energy ball milling (HEBM) in ball mills is an effective method for obtaining metallic alloys, including multicomponent “high-entropy” alloys [5–8]. Typically, after HEBM, the powder product, grinding balls, and milling drums heat up by only a few dozen degrees Celsius, and in highly energy-intensive mills, water cooling of the drums is used. Therefore, during mechanical alloying, mutual dissolution of metals occurs at relatively low temperatures, where diffusion coefficients in solid metals are very low and cannot explain the formation of a homogeneous alloy within the experimentally observed time frames of a few minutes to several hours. However, it is possible that the local temperature at grinding ball contact points significantly exceeds the average temperature. Currently, it is not feasible to measure the temperature at contact points directly during HEBM, but no signs of melting in particles after mechanical processing were found in our experiments. Thus, even at friction contact points, the temperature remained below the lowest melting temperature of the components in this system (1084 °C for copper). It is generally accepted that mutual friction and deformation of metallic particles significantly accelerate solid-state diffusion processes, similar to the acceleration of polymerization under shear deformation conditions [9]. The possibility of ultrafast mutual dissolution of metals due to intense deformation has been demonstrated using molecular dynamics simulations [7]. Several studies have examined the deformation of individual metallic powder particles during HEBM [10–14], while other research has focused on direct observation (video recording) of the motion of grinding balls in the drum [15–16]. However, experimental data on the dynamics of mutual metal dissolution during HEBM in ball mills remain scarce.

Nickel and copper occupy adjacent positions in the Periodic Table of Elements, with atomic numbers 28 and 29, respectively. They share the same face-centered cubic (FCC) crystal structure with relatively close unit cell parameters ($a_{\text{Ni}} = 3.524 \text{ \AA}$, $a_{\text{Cu}} = 3.615 \text{ \AA}$), allowing easy phase differentiation in

X-ray diffraction patterns. The atomic radii of these elements are also very similar: $R_{\text{Ni}} = 1.15 \text{ \AA}$, $R_{\text{Cu}} = 1.17 \text{ \AA}$. Due to these properties, Ni and Cu exhibit unlimited mutual solubility in the solid state, forming a continuous series of solid solutions. Thus, the Ni–Cu system is well suited for studying the process of mutual dissolution of solid metals, i.e., atomic interpenetration during HEBM.

The objective of this study is to investigate the mixing and mutual dissolution processes of copper and nickel powders depending on the planetary mill operating conditions, gas atmosphere inside the milling drums, and processing duration. Initially, the influence of processing parameters in the Aktivator-2S planetary mill (rotational speed of the main disk and milling drum) and the duration of HEBM on powder structure in an air atmosphere is examined to determine the most efficient mode. Next, the effect of the gas atmosphere is studied under the identified optimal conditions. Thus, the study aims to determine HEBM conditions that enable the fastest mutual dissolution of metals, which is important for developing mechanosynthesis methods for metallic alloys, including high-entropy alloys.

2. Materials and Methods

2.1. Initial components

The starting materials were an equimolar mixture of nickel powder PNE-1 with a particle size of $d = 45\text{--}71 \text{ \mu m}$ and a purity of 99.5 % (Russian Standard 9722-97) and copper powder PMS-1 with $d = 50\text{--}70 \text{ \mu m}$ and a purity of 99.5 % (Russian Standard 4960-75). Ni and Cu powders were mixed in equal molar proportions (Cu : Ni = 1 : 1) for 2–3 minutes before being placed into the drums of the Aktivator 2S mill (Aktivator, Russia).

2.2. Method for preparing metallic alloys

The pre-mixed batch was loaded into the activator drums along with 6 mm diameter steel grinding balls in a mass ratio of 20 : 1 (200 g of balls per 10 g of mixture). The powder mixture underwent high-energy ball milling for durations ranging from 5 to 90 minutes at speeds of 300, 500, and 694 rpm. HEBM in argon, nitrogen, helium, or air atmospheres

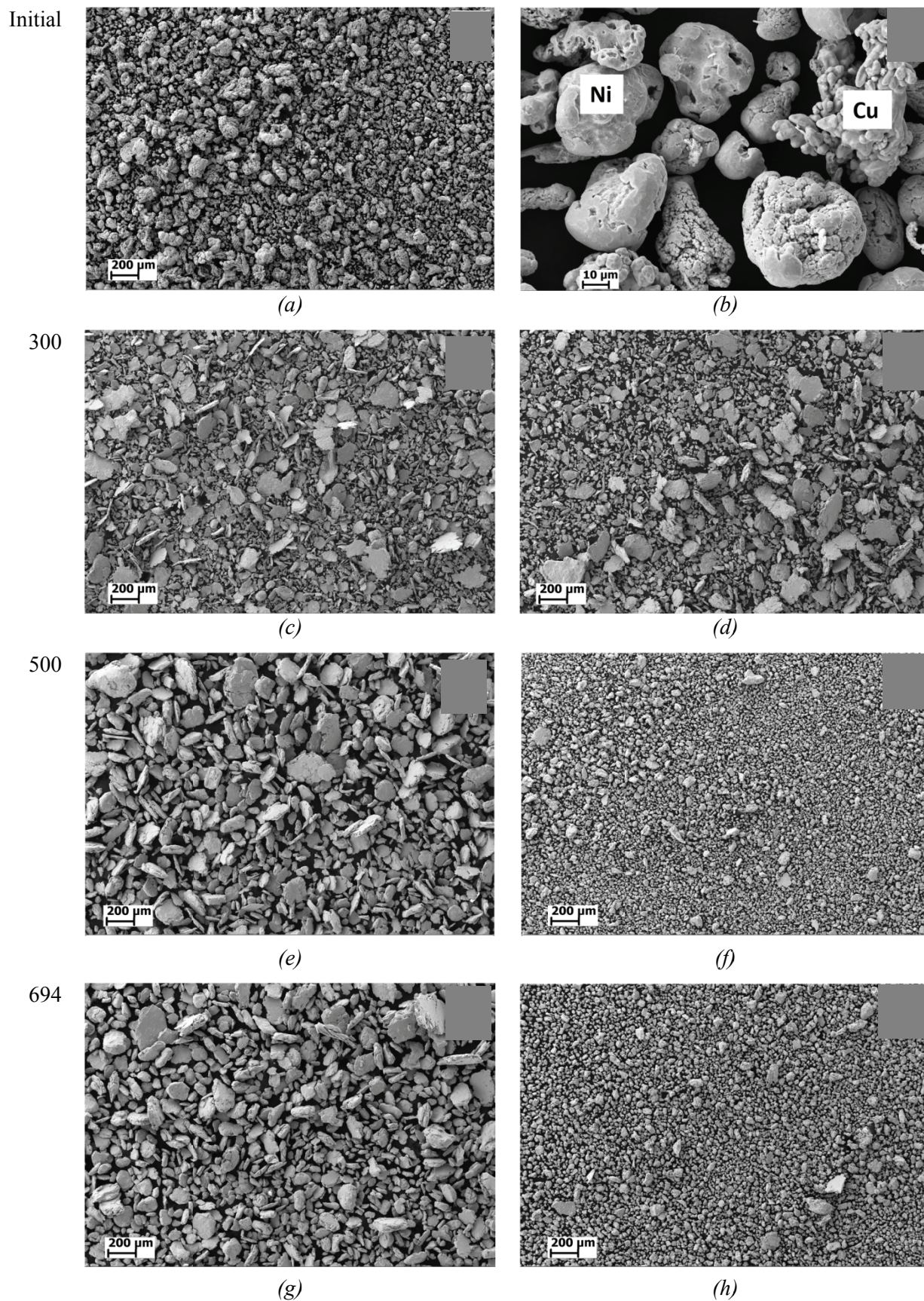


Fig. 1. Photograph of the microstructure of the initial mixture (a, b) and mixtures after HEBM for 30, 90 min 300 rpm (c, d), 30, 90 min 500 rpm (e, f), 30, 90 min 694 rpm (g, h) in air

was conducted at 694 rpm for 90 minutes. The milling drums were hermetically sealed with lids equipped with valves for gas evacuation and introduction. Initially, vacuuming was performed down to a residual pressure of 0.01 Pa, followed by filling the drums with gas to a pressure of 0.6 MPa. In all experiments, the ratio of drum rotation speed to the main disk rotation speed remained constant at $K = 2$.

2.3. Analytical methods

Studies were conducted to analyze the phase composition, surface morphology, and crystal structure of the obtained nickel and copper powder mixtures. The research utilized X-ray phase analysis (XRD) on a DRON-3M diffractometer and scanning electron microscopy (SEM) on a Zeiss Ultra+ microscope (Carl Zeiss, Germany) with energy-dispersive spectroscopy (EDS).

3. Results and Discussion

Figure 1 presents micrographs of the Ni + Cu mixture before and after HEBM in an air atmosphere. The initial powder particles exhibit a dendritic morphology typical of metals obtained via the electrolytic method, with nickel particles being more rounded and copper particles showing a more pronounced dendritic structure (Fig. 1a, b). The particle size does not exceed 100 μm , with the particles forming conglomerates of smaller dendritic elements ranging from 5 to 10 μm .

After HEBM under relatively mild conditions at 300 rpm, the particles flatten into thin flakes measuring up to 200–300 μm (Fig. 1c, d). This morphology is maintained for up to 90 minutes of processing. Increasing the milling speed to 500–694 rpm and processing for 30 minutes leads to an increase in flake thickness (Fig. 1e, g). Extension of the HEBM duration to 90 minutes results in the fragmentation of flakes and the formation of smaller particles with both irregular and rounded shapes (Fig. 1f, h).

The images indicate that the increase in flake thickness occurs due to the adhesion of primary flakes to one another, forming multilayered particles. At the fragmentation stage, the bulk of the powder consists of uniform, rounded particles ranging from 10 to 50 μm in size.

The X-ray phase analysis showed that at a milling speed of 300 rpm, no mutual dissolution of Ni and Cu occurs even after the maximum processing duration of 90 minutes – all diffraction patterns display only the peaks of the initial metals (Fig. 2a). At a speed of 500 rpm, the peaks of the initial metals

remain present during the first 30 minutes, gradually broadening while their intensity decreases. However, they eventually disappear completely, giving way to broad peaks corresponding to the NiCu solid solution (Fig. 2b).

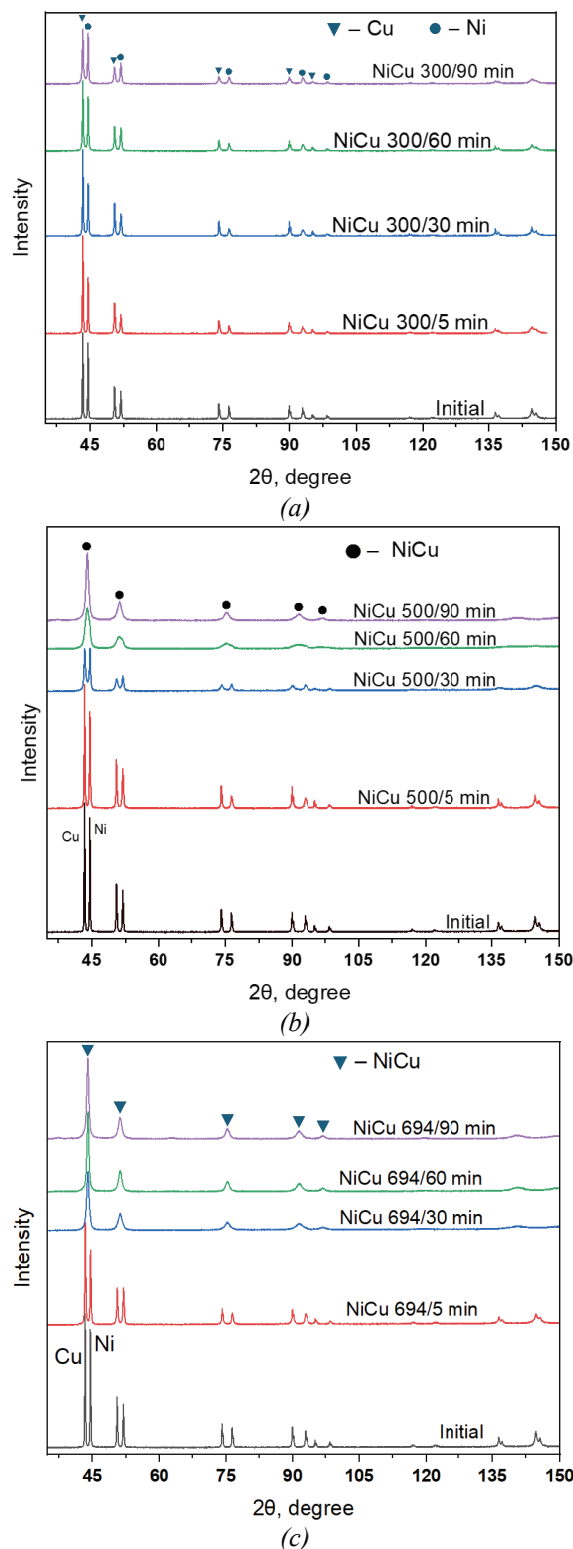


Fig. 2. Diffraction patterns of the Ni + Cu mixture after HEBM in air under different processing conditions: 300 rpm (a), 500 rpm (b) and 694 rpm (c)

At a milling speed of 694 rpm, the peaks of the initial materials remain visible after 5 minutes of processing. The NiCu solid solution phase forms between 5 and 30 minutes of HEBM (Fig. 2c). As the processing time increases, the peak widths gradually expand while their intensity decreases. Eventually, the peaks of the initial metals disappear completely, giving way to broad peaks corresponding to the NiCu solid solution (Fig. 2c), similar to Fig. 2b.

Notably, none of the X-ray diffraction patterns simultaneously show peaks of both the initial metals and the solid solution. This suggests that alloying occurs rapidly throughout the entire powder volume once a critical state of the mixture is reached. The solid solution exhibits an FCC structure with a lattice parameter of $a_{\text{NiCu}} = 3.570215(41) \text{ \AA}$, which

falls between the lattice parameters of the original metals, in accordance with Vegard's law.

The microstructural analysis and elemental distribution studies confirmed that HEBM in an air atmosphere results in the formation of a homogeneous solid solution (Fig. 3).

To study the effect of the gas atmosphere, the Ni + Cu mixture was processed continuously for 90 minutes at 694 rpm, with $K = 2$ (where K is the ratio of the rotational speeds of the milling jars and the planetary disc), in atmospheres of air, Ar, N₂, and He. The results showed that the shape and size of the metallic particles are highly dependent on the gas atmosphere inside the milling jar, as well as the continuity of the process (Fig. 4).

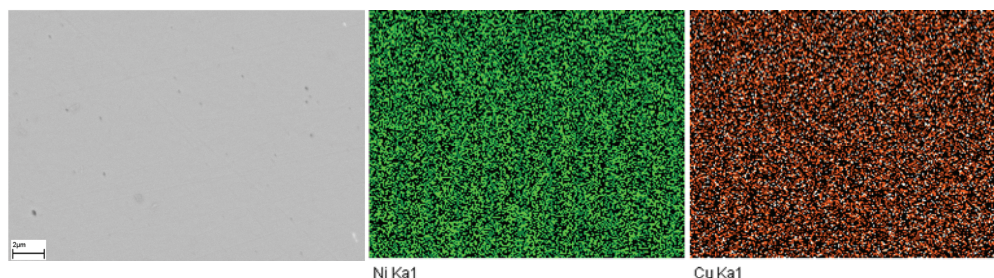


Fig. 3. Microstructure (SEM) and element distribution maps (EDM) after HEBM in air

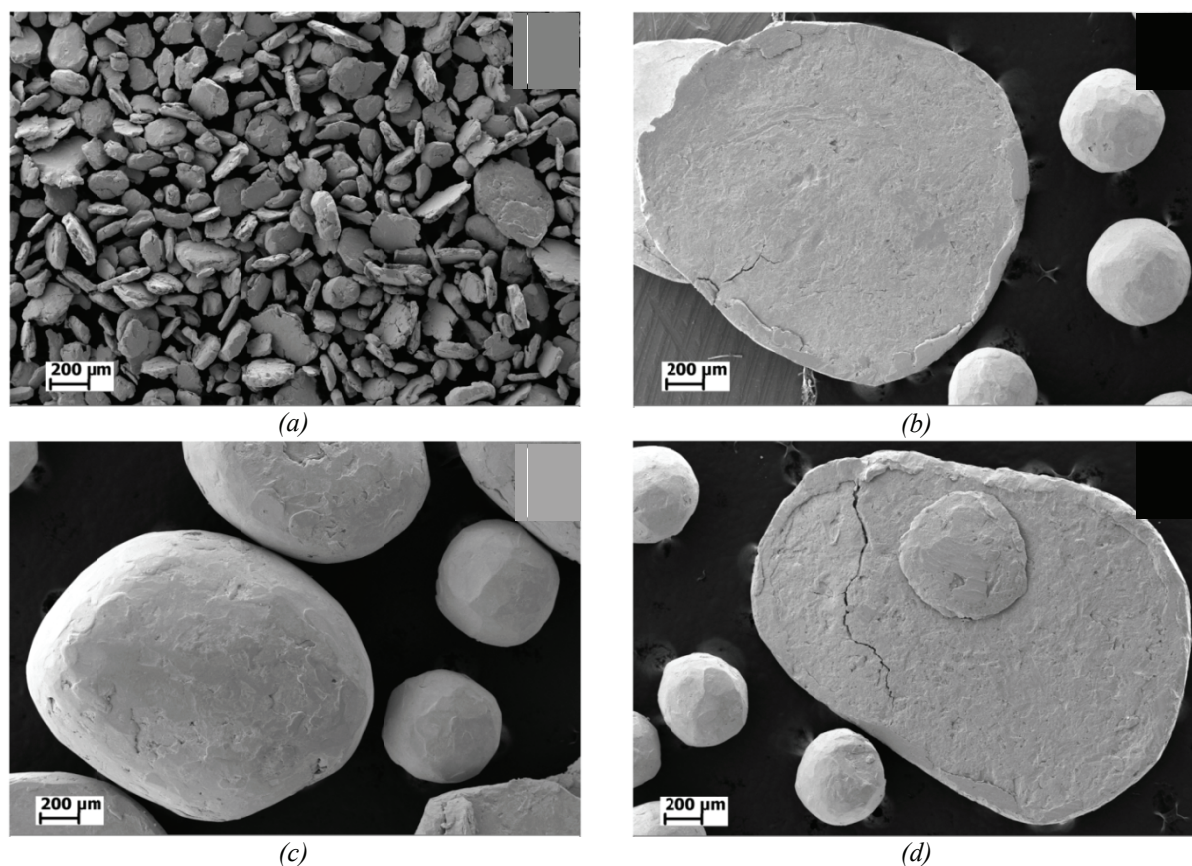


Fig. 4. Photograph of the microstructure of mixtures after HEBM for 90 min at 694 rpm in an atmosphere of air (a), Ar (b), N₂ (c), He (d)

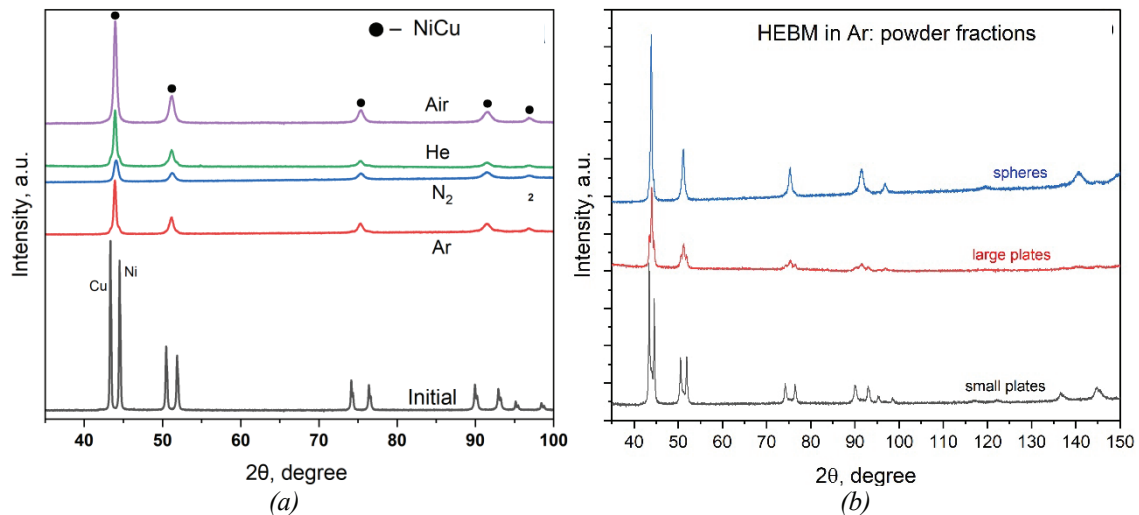


Fig. 5. Diffraction patterns of a Ni + Cu mixture after HEBM for 90 min at 694 rpm in different atmospheres (a), and particles of different shapes after treatment in argon (b)

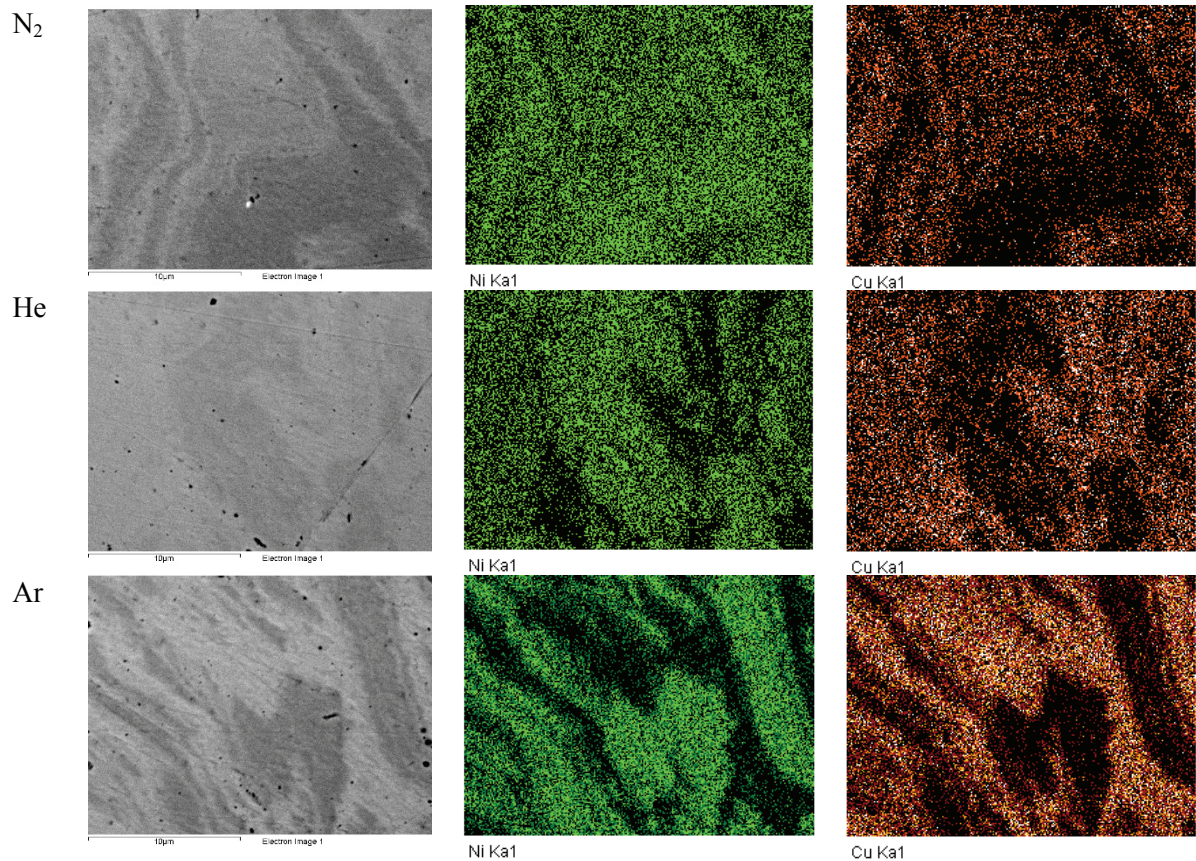


Fig. 6. Microstructures and element distribution maps in particles formed after treatment of Ni + Cu mixtures in Ar, N₂ and He at 694 rpm for 90 min

The powder processed continuously in an air atmosphere for 90 min consists of uniformly sized flakes (Fig. 4a), in contrast to the powder processed for 90 min with periodic stops and depressurization of the milling jar at 5, 30, 60, and 90 min (Fig. 1h). This suggests that each time the jar is opened, partial oxidation of the processed powder occurs, leading to

a gradual accumulation of oxygen. As a result, powders processed with interruptions become more brittle.

The particle size and morphology of powders processed in oxygen-free atmospheres differ significantly from those processed in air, as shown in Figs. 4b, c, d. These powders consist of disk- and

oval-shaped flakes, as well as spherical particles, with the spherical particles measuring approximately 500–700 μm , while the longitudinal size of the flakes reaches 2–3 mm. This significant increase in particle size indicates that cold welding processes are drastically accelerated in oxygen-free atmospheres.

The results of the X-ray phase analysis, presented in Fig. 5a, show that the primary phase after HEBM is always the NiCu solid solution with an FCC structure. In samples processed in He or Ar, weak peaks of the initial metals are observed alongside the main solid solution phase (appearing as slight protrusions on the left and right sides of the primary phase peaks).

A more detailed analysis was conducted for the sample processed in Ar (Fig. 5b). This powder was separated into fractions based on particle shape: small flakes, large flakes, and spherical particles. Each fraction was individually analyzed using X-ray phase analysis. The results revealed that small flakes consist mainly of the original metals, large flakes contain diffraction peaks of both the solid solution and the

initial components, while in spherical particles, the solid solution dominates.

Elemental distribution inhomogeneities are visible in the cross-sections of powder particles, as shown in Fig. 6. Regions enriched with Ni or Cu appear as elongated bands, indicating intense plastic flow of metals during HEBM.

The microsection analysis of all samples at different magnifications revealed typical microstructural inhomogeneities caused by incomplete mutual dissolution of metals, as shown in Fig. 7. As seen in the figure, no inhomogeneities are observed when processing in an air atmosphere. However, in oxygen-free atmospheres, both homogeneous regions and inhomogeneous layered regions coexist, with the layers displaying a highly irregular, wavy structure.

The quantitative X-ray spectral microanalysis (EDS) revealed that homogeneous regions have a composition close to the target equiatomic NiCu solid solution.

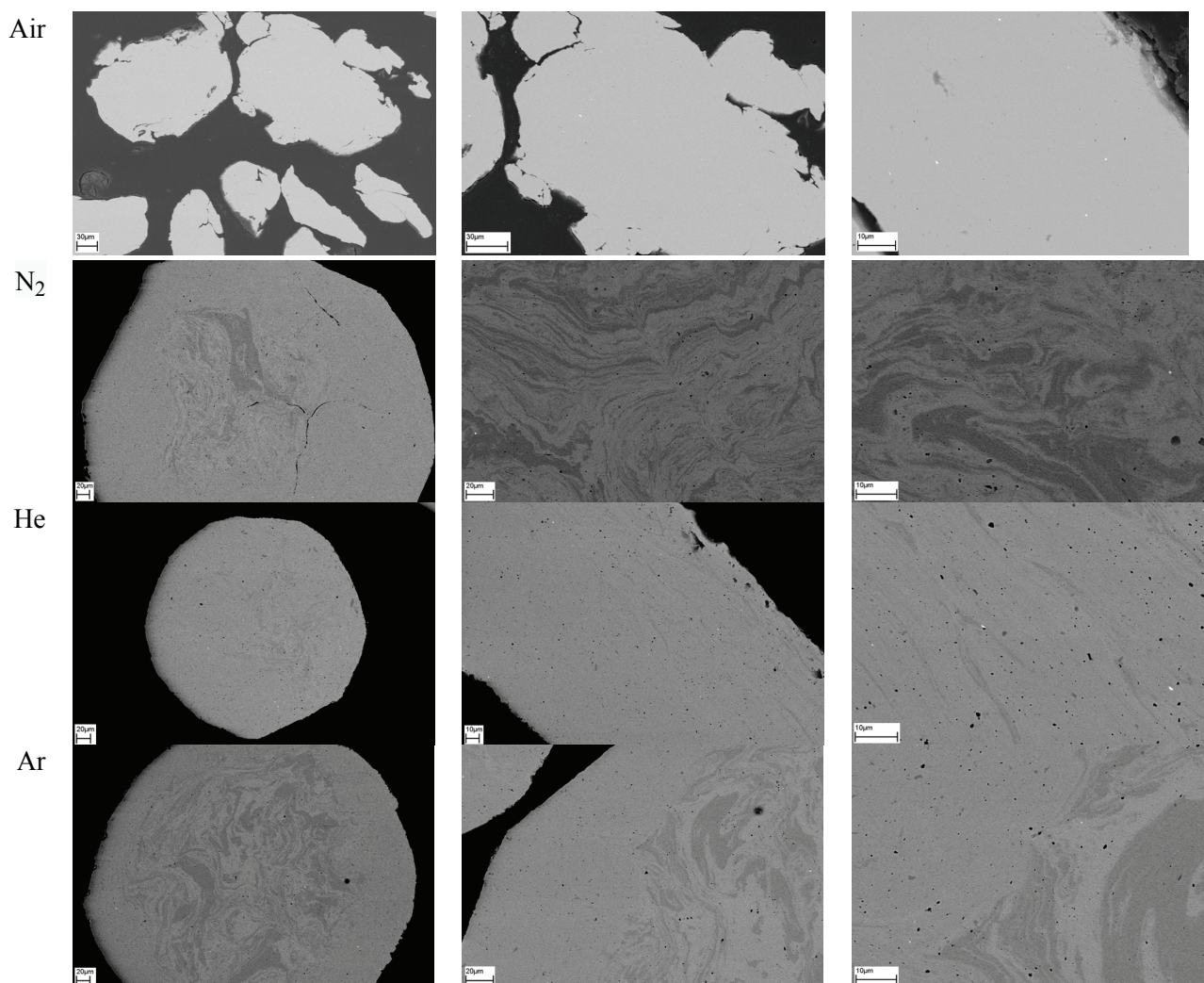


Fig. 7. Microstructures of particles obtained in different atmospheres after HEBM for 90 min at 694 rpm

Inhomogeneous regions show darker layers enriched in nickel and lighter layers enriched in copper. The darkest areas correspond to nearly pure nickel, while the light areas exhibit a wide range of compositions, from pure copper to the equiatomic NiCu solid solution.

4. Conclusion

The mutual dissolution of metals during HEBM occurs due to the flow of metallic layers and their friction against each other. For this to happen, the mechanical stresses induced by the grinding balls on the metal particles must exceed the yield strength of the metals. In mild planetary mill conditions (e.g., 300 rpm), this threshold is not reached, and even prolonged processing does not lead to the formation of a solid solution. Critical stresses are generated at a milling speed of 500 rpm, where a solid solution forms after 1 hour of processing in an air atmosphere. Further increasing the milling speed accelerates the process – at 694 rpm, the solid solution forms within 5 to 30 minutes of HEBM.

At the initial stages of HEBM, particles flatten into plate-like (flake-like) structures. In an oxygen-free atmosphere, intense cold welding of these flakes occurs, forming large (2–3 mm) disc-shaped and oval plates. These plates then undergo rounding, eventually acquiring a spherical shape. In an air atmosphere, oxidation of the metal particle surfaces slows down cold welding, and the metals become less ductile due to oxygen absorption. The HEBM in air results in the powder consisting of smaller particles with a rounded and plate-like morphology (300–400 μm along the plate surface, less than 100 μm across). The depth of shear stress impact, which drives the flow of metallic layers within the particles, is approximately 100 μm . Therefore, in the core of large particles formed during HEBM in an inert atmosphere, unreacted layers of initial reagents and intermediate solid solutions remain. Thus, the optimal regime for the mechanosynthesis of metallic solid solutions is planetary milling at speeds above 500 rpm in an air atmosphere. These findings can be used to optimize the mechanical synthesis of metallic solid solutions, including Cantor high-entropy alloys with an FCC structure.

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6. Conflict of interest

The authors declare no conflicts of interest.

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