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Ön Alaşımlı NiTi Tozlarına Cu İlavesinin Mikroyapısal Etkileri

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ÖZ

Bu çalışmada, $(NiTi)_{95}Cu_5$ ağırlıkça % alaşım tozları, ön-alaşımlı NiTi tozuna Cu ilavesi yapılarak 60, 120 ve 180 dakika sürelerle mekanik alaşımlama yöntemiyle üretilmiştir. Öğütme süresinin morfoloji, elementel dağılım, partikül boyutu ve faz evrimi üzerindeki etkileri sistematik olarak incelenmiştir. Tozlar SEM, EDS, partikül boyut analizi ve XRD teknikleri kullanılarak karakterize edilmiştir. SEM gözlemleri, tozların genel olarak küresel bir morfolojiyi koruduğunu, ancak daha uzun öğütme süreleriyle yüzey deformasyonu ve aglomerasyonun arttığını ortaya koymuştur. EDS sonuçları, tüm numunelerde Ni, Ti ve Cu elementlerinin varlığını doğrulamış ve öğütme süresi arttıkça Cu dağılımının daha homojen hale geldiğini göstermiştir. Partikül boyut analizi, ortalama partikül boyutunun belirgin biçimde azalmadığını, buna karşın boyut dağılımının genişlediğini göstermiştir. XRD sonuçları, NiTi, Ti(Ni,Cu), Ni_4Ti_3 ve Ni_3Ti fazlarının varlığını ortaya koymuştur. Öğütme süresi arttıkça, Ni_3Ti ve Ni_4Ti_3 fazlarına ait göreli pik şiddetlerinin azaldığı, buna karşılık NiTi esaslı faz yapısının daha baskın hale geldiği belirlenmiştir. Bu durum, daha homojen bir NiTiCu ilişkili faz yapısına doğru ilerleyen bir dönüşüme işaret etmiştir. Sonuç olarak, mekanik alaşımlama süresinin Cu ile modifiye edilmiş NiTi sistemlerinde faz evrimi, elementel homojenlik ve parçacık morfolojisinin kontrolünde belirleyici bir rol oynadığı belirlenmiştir.

Microstructural Effects of Cu Addition on Pre-Alloyed NiTi Powders

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ABSTRACT

In this study, $(NiTi)_{95}Cu_5$ wt% alloy powders were produced by mechanical alloying of pre-alloyed NiTi powder with Cu addition for 60, 120, and 180 minutes. The effects of milling duration on the morphology, elemental distribution, particle size, and phase evolution were systematically investigated. The powders were characterized using SEM, EDS, particle size analysis, and XRD techniques. SEM observations revealed that the powders maintained a generally spherical morphology, while surface deformation and agglomeration increased with longer milling times. EDS results confirmed the presence of Ni, Ti, and Cu elements in all samples, with Cu distribution becoming more homogeneous as the milling time increased. Particle size analysis indicated that the average particle size did not decrease significantly; however, the size distribution became broader. XRD results showed the presence of NiTi, Ti (Ni,Cu), Ni_4Ti_3, and Ni_3Ti phases. With increasing milling time, the relative intensities of Ni_3Ti and Ni_4Ti_3 phases decreased, while the NiTi-based phase structure became more dominant, indicating a progressive shift toward a more homogeneous NiTiCu-related phase constitution. Overall, it was found that the mechanical alloying time plays a decisive role in controlling phase evolution, elemental homogeneity, and particle morphology in Cu-modified NiTi.

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1. Introduction

Shape memory alloys have attracted considerable attention in scientific and technological fields in recent years because of the smart and functional properties they show. These alloys exhibit shape memory effect (SME - ŞHE) and superelasticity (SE) behavior. The main reason for this behavior comes from a two-way mechanism called martensitic transformation (MT). These mechanisms are defined by characteristic transformation temperatures where the alloys change to the martensite phase during cooling (Ms: martensite start) and to the austenite phase during heating (As: austenite start) (Otsuka and Ren, 1999).

Shape memory alloys can generally be grouped into NiTi-based, Cu-based, and Fe-based systems, together with high-temperature shape memory alloys developed for more demanding service conditions. Among these groups, Cu-based alloys such as Cu–Zn–Al and Cu–Al–Ni are often considered attractive because of their relatively low cost, although their mechanical stability is still limited. Fe-based alloys have also drawn attention, particularly for large-scale and cost-sensitive engineering applications. However, their shape memory performance generally remains below that of NiTi-based alloys (Amadi et al., 2024; Hossain et al., 2025). In NiTi-based systems, Cu addition is frequently considered as a useful alloying strategy for tailoring transformation behavior. For example, Cu addition offers the potential to decrease the transformation temperatures while increasing the thermal stability of NiTiHf-based alloys (Acar, 2018). However, it has also been reported that excessive Cu concentration may lead to a risk of losing the shape memory property (Dağdelen et al., 2017).

Among these materials, nickel-titanium (NiTi)-based alloys (Nitinol) are considered among the most functionally important shape memory alloy systems (Nakata et al., 1999; Otsuka and Ren, 2005). NiTi alloys exhibit a clear difference from other SMA systems in various aspects, especially martensitic crystal structure, elastic and mechanical properties. The austenite (B2 cubic) phase of NiTi is stable at high temperatures, while the martensite phase is generally in a monoclinic (B19') structure and has a lower lattice structure. In some NiTi alloys, the transformation from B2 phase to B19' phase happens directly, while in some alloys it can happen first through an intermediate phase called R-phase (rhombohedral) (Zhang and Sehitoglu, 2004; Shamimi et al., 2018).

NiTi alloys offer a wide range of applications due to the superior combination of properties they have. These properties include excellent corrosion resistance, good mechanical strength, superelastic behavior, shape memory, and good biocompatibility (Jabur et al., 2013). These alloys have become a favorite in their field besides biomedical applications, they have the potential to be used as functional materials in many engineering applications such as active and adaptive structures or smart structures (Bram et al., 2002).

Traditionally, SMAs are generally produced by vacuum arc or induction melting and hot forming processes (Mwangi et al., 2019). However, casting methods can create problems such as a heterogeneous structure, impurity contamination like oxygen or carbon coming from the crucible, and segregation (Khanlari et al., 2018; Salvert et al., 2018). Considering the high reactivity and high melting temperatures of NiTi alloys, Powder Metallurgy (PM - TM) techniques offer attractive alternatives to overcome these challenges (Rostami et al., 2019). Powder metallurgy methods offer the ability to produce controlled porous structures (needed for biomaterials) and the potential to keep impurity levels low (Bram et al., 2002).

Among powder metallurgy techniques, mechanical alloying (MA) remains an important and versatile method for the production of nano-structured and amorphous materials (Novák et al., 2015; Yuan et al., 2024; Suryanarayana and deBarbadillo, 2025).

Mechanical alloying (MA) performed in high-energy ball mills allows obtaining alloys with a fine microstructure as a result of the repeated cold welding and fracturing movements of the powders continuously (Ye et al., 1998). An increase in alloying time may cause the material to become amorphous and leads to unwanted contaminations. Therefore, short-time mechanical alloying is important for the rapid synthesis of intermetallic compounds like TiNi (Maziarz et al., 2004; Nobuki et al., 2019).

The main factor that determines the performance of shape memory alloys in practical applications is the austenite–martensite phase transformation temperatures of the alloy. To control these temperatures and make them specific to the application, adding third elements to the NiTi base alloy is a common strategy. Copper (Cu) is an important alloying element used for this purpose (Behvar et al., 2025; Hossain et al., 2025).

Literature studies show that Cu addition to the NiTi alloy system generally decreases the transformation temperatures significantly. For example, in $(NiTi)_{100-x}Cu_x$ alloys, increasing Cu content was reported to lower the onset of martensitic transformation from about 70 °C to nearly −60 °C (Abdelhady et al., 2025).

In another recent study, even 1 at.% Cu addition led to a reduction in transformation temperatures of more than 80 °C (Shen et al., 2025). These findings indicate that Cu addition is an effective way to shift NiTi alloys toward lower operating temperatures. However, excessive Cu addition may also alter the phase constitution and adversely affect the shape memory behavior.

While efforts to optimize the performance of NiTi alloys continue, the effects of mechanical alloying process parameters, particularly at short milling times, on the microstructural evolution of Cu-added pre-alloyed NiTi powders have not yet been fully discussed in the literature. In particular, the influence of milling time on phase structure, elemental distribution, and particle morphology deserves further attention. Therefore, this study investigates the effect of mechanical alloying (MA) time (60, 120, and 180 min) on these key microstructural features in Cu-added pre-alloyed NiTi powders. The use of pre-alloyed NiTi powder also allows the study to focus more directly on the role of Cu addition and milling time. In this way, the study aims to contribute to the identification of suitable short-time processing conditions and to provide a clearer understanding of microstructural evolution in this material system.

2. Material and Method

The experimental flow of the study is shown graphically in Figure 1.

2.1. Powder preparation and mechanical alloying process

In the experiments, pre-alloyed NiTi powder (Ni50Ti50, supplied by Yuanshan Advanced Materials Co., Ltd.) and pure copper powder were used as starting materials in ($Ni_{50}Ti_{50}$) atomic ratio. The purity of the NiTi powder is >98.5%, and the average particle size is <50 µm. The purity of the copper powder is >99%, and the average particle size is <44 µm. SEM and XRD images of the powders are seen in Figure 2. Cu addition was mixed to make up 5 wt % of the total mixture. The codes of the Cu-added powder samples are given in Table 1. Here, NT0 denotes the as-received pre-alloyed NiTi powder used as the reference sample (without Cu addition and milling). In the following sections, the samples will be expressed using these codes.

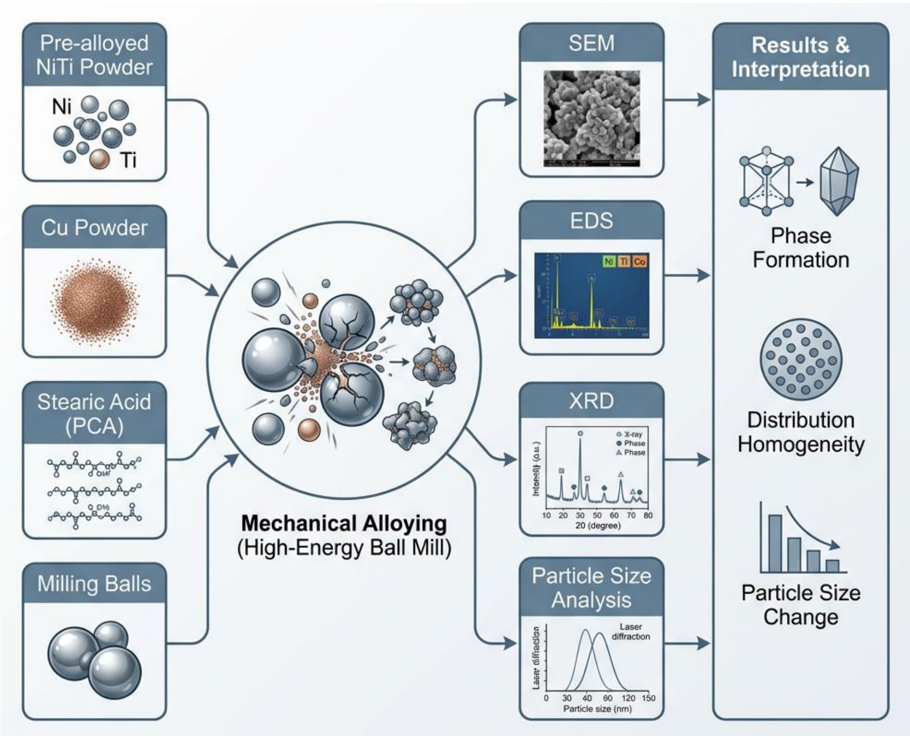

Figure 1. Graphical flow diagram of the experimental process

The samples were placed into stainless steel containers by adding Ø5 mm stainless steel balls so that the ball-to-powder ratio was 5:1. To obtain a homogeneous mixture and prevent agglomeration, 2% stearic acid was used. Before milling, the lids were closed in a leak-proof way. The milling process was carried out using a high-energy ball mill, Fritsch Pulverisette 7. The powder mixtures were milled in “reverse mode” at a constant speed of 300 rpm with a “30 min run - 5 min rest” cycle. The process was completed by applying different milling times of 60, 120, and 180 minutes. These milling times were selected based on the literature and were intended to represent short-time mechanical alloying conditions relative to the longer durations commonly reported. In this way, the study aimed to evaluate whether noticeable changes in phase structure, elemental distribution, and particle morphology could already be observed at the early stages of milling.

Table 1. Chemical compositions of the powders (wt%)

Sample code	Composition symbolic	Composition explanation	Milling time (min)
NT0	Pre-alloyed NiTi	Pre-alloyed NiTi	-
NTC1	$(NiTi)_{95}Cu_5$	Pre-alloyed $Ni_{50}Ti_{50}$ + 5 wt% Cu	60
NTC2	$(NiTi)_{95}Cu_5$	Pre-alloyed $Ni_{50}Ti_{50}$ + 5 wt% Cu	120
NTC3	$(NiTi)_{95}Cu_5$	Pre-alloyed $Ni_{50}Ti_{50}$+ 5 wt% Cu	180

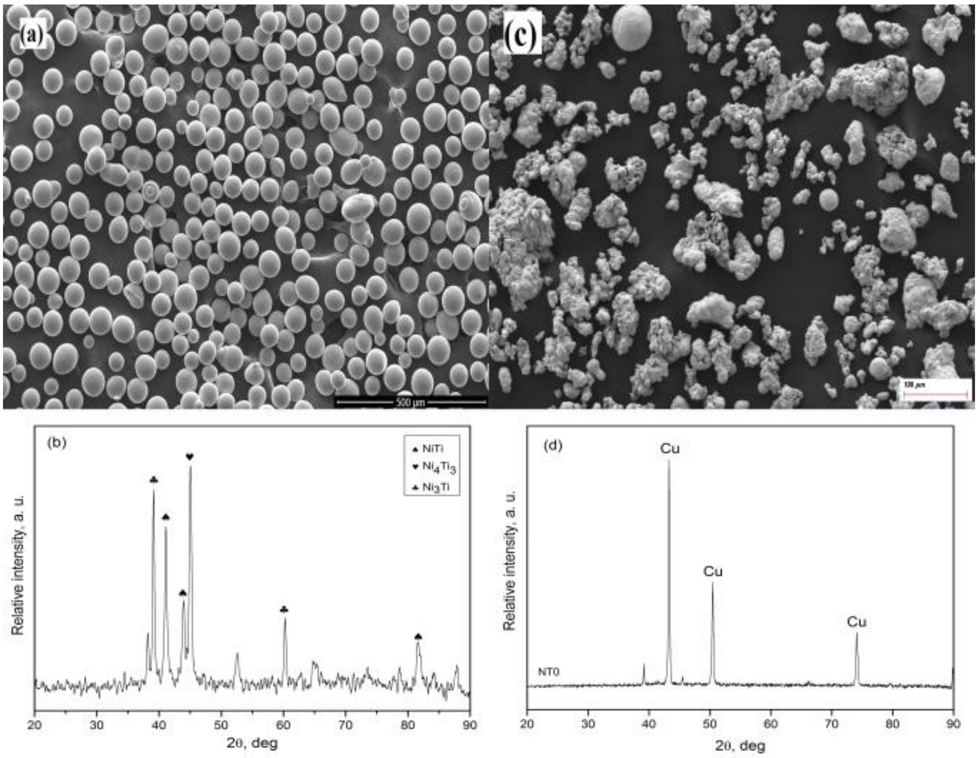

Figure 2. Characterization results of the starting powders: (a) SEM and (b) XRD for the pre-alloyed NiTi powder; (c) SEM and (d) XRD for the Cu powder

2.2. Experimental methods and analysis

To examine the microstructural properties of the powders, a ZEISS GEMINI SEM 560 Scanning Electron Microscope with high-resolution capability was used. This device made it possible to image the surface topography, morphological properties, and particle distributions of the powders in detail. To determine the chemical composition of the samples, EDS (Energy Dispersive X-ray Spectroscopy) integrated into the microscope was used, and this analysis allowed identifying the types of elements in the powders and their approximate amounts.

To determine the particle sizes of the powders, measurements were made on SEM images. For determining particle sizes, measurements were carried out on multiple SEM images taken from different regions. The measurement and image processing steps were performed using Fiji (ImageJ) software. As a result, the size of the particles was measured and distribution data were obtained.

A Bruker D8 Advance X-ray diffractometer (XRD) was used to define the changes in the crystal phase composition of the samples and to reveal the distribution of phases. The analyses were carried out at room temperature using a Cu Kα radiation source (λ = 1.5406 Å) in the 20°– 90° 2θ range, with a scanning speed of 2°/min. These analyses are critical for detecting possible new intermetallic phases that may form with Cu addition, as well as the characteristic austenite (B2) and martensite (B19') phases of NiTi.

3. Results and Discussion

In Figures 3-5, the morphological, chemical, and size distribution results of powders with the same composition that were subjected to mechanical alloying for different times (60, 120, and 180 min) are given. According to the SEM images (Figures 3a-5a), in all samples the powders generally show a morphology close to spherical form, with smooth surfaces and a homogeneous distribution. However, as the mechanical alloying time increases, it was observed that the effect of cold welding between particles becomes more evident, and especially in NTC3 some powders show a partial tendency to agglomeration (Zeybek, 2025).

When the particle size analyses are examined (Figures 3b-5b), the average particle size was measured as 50.9 µm for NTC1, 51.6 µm for NTC2, and 61.1 µm for NTC3. Compared with the starting powders, the average particle size showed an increasing trend. Although no clear reduction in average particle size was observed, the standard deviation increased from 13.96 µm for NTC1 to 16.24 µm for NTC2 and 37.57 µm for NTC3, indicating a progressive broadening of the particle size distribution with increasing milling time. This suggests that fragmentation and agglomeration occurred simultaneously during mechanical alloying, and that the system did not reach a stable balance. Similarly, Cîrstea et al. (2024) reported that, in NiTiCu alloys, mechanical and thermal processing lead to a broader particle size distribution rather than particle refinement, and that the optimum processing time is critical for achieving structural homogeneity.

According to the EDS maps (Figures 3c-e, 4c-e, 5c-e), while Ni and Ti elements show a homogeneous distribution in all samples, it was seen that the Cu element is concentrated especially in NTC1 in more local and clustered regions. In NTC2 and NTC3, the Cu distribution became more balanced, and in the elemental maps Cu is seen to spread better into the matrix. This indicates that with increasing mechanical alloying time, Cu shows more effective mixing/dispersion within the NiTi matrix. Similarly, Chen et al. (2024); reported in NiTiCu alloys produced by the twin-wire arc AM method that the uniform distribution of Cu into the matrix increases the phase transformation stability of the material. Also, in the SLM-based study conducted by Ba et al. (2024); it was stated that the homogeneous distribution of Cu content into the NiTi matrix strengthens the stability of the B2 phase and contributes to the decrease of Ti_3Ni_4 precipitates.

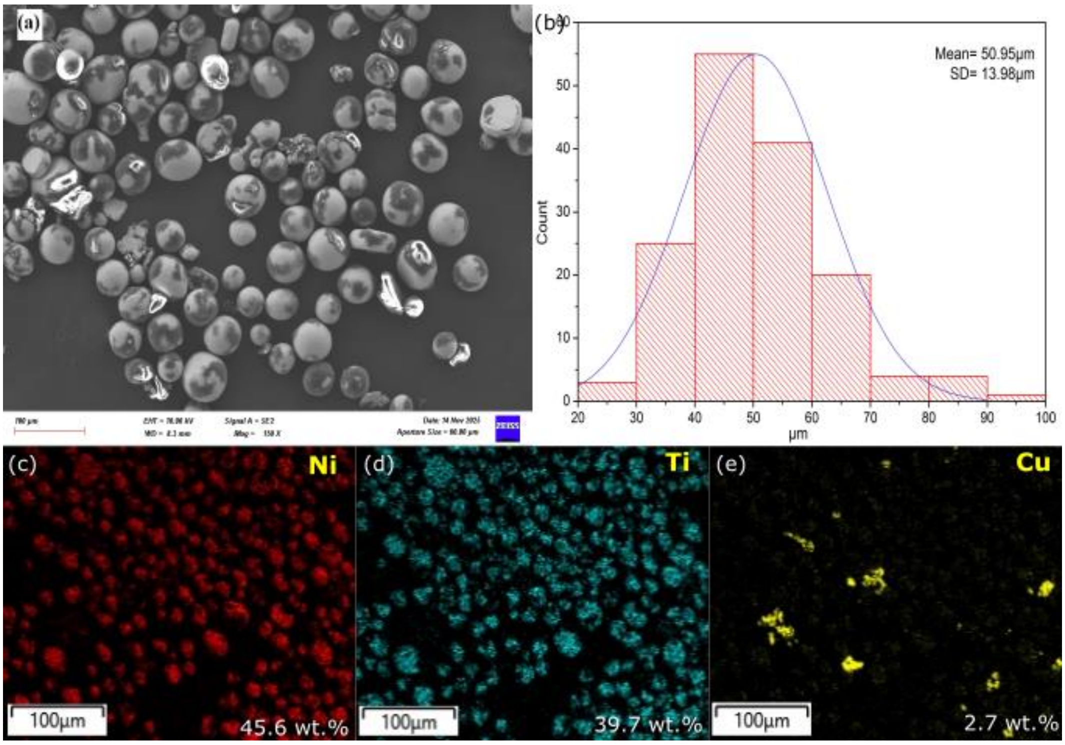

Figure 3. (a) SEM image, (b) particle size distribution, and (c-e) elemental maps of Ni, Ti, and Cu for the NTC1 sample

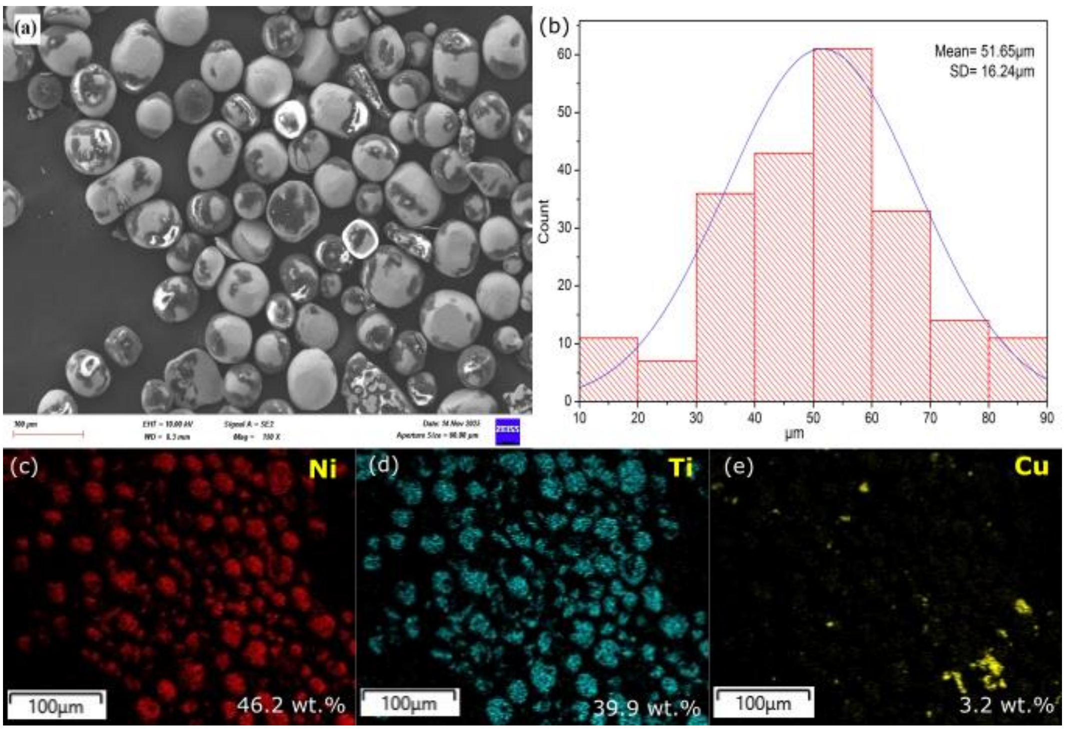

Figure 4. (a) SEM image, (b) particle size distribution, and (c-e) elemental maps of Ni, Ti, and Cu for the NTC2 sample

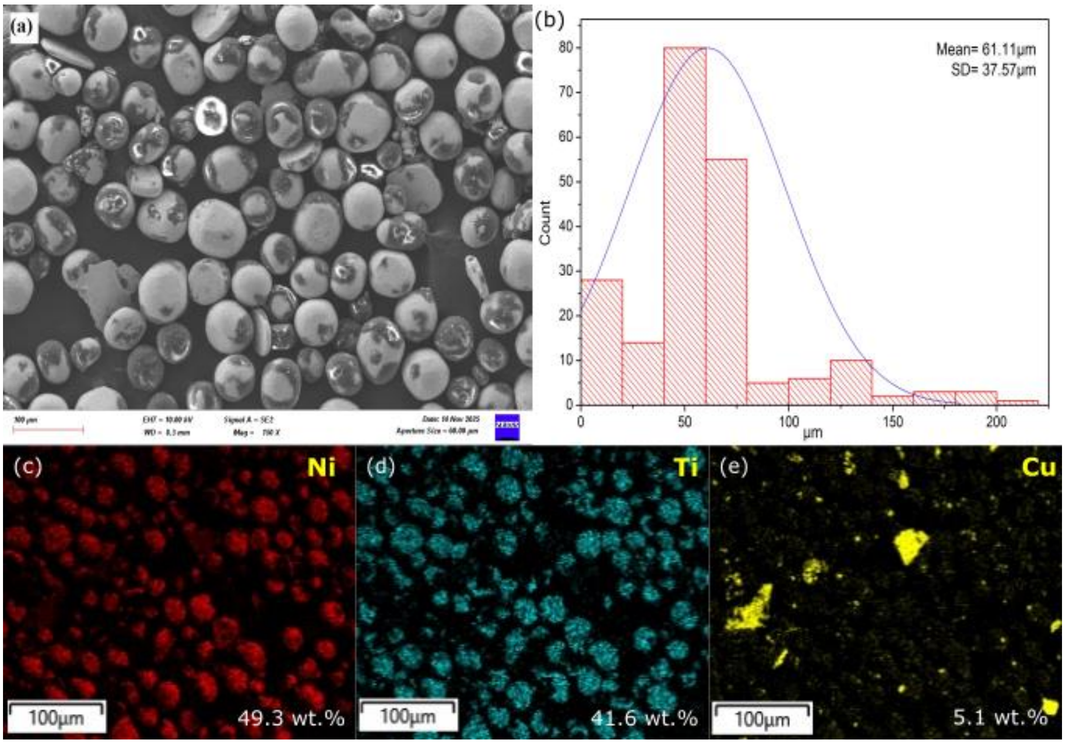

Figure 5. (a) SEM image, (b) particle size distribution, and (c-e) elemental maps of Ni, Ti, and Cu for the NTC3 sample

Although the nominal composition is $(NiTi)_{95}Cu_5$ (wt.%), in the EDS results the Cu ratio was measured as 2.7 wt% (NTC1), 3.2 wt% (NTC2), and 5.1 wt% (NTC3), respectively. This difference may be attributed not only to material-related factors, but also to the limitations of the measurement method. From a materials perspective, the lower Cu values obtained for NTC1 and NTC2 may be associated with the non-uniform distribution of Cu within the powder particles during short-time mechanical alloying. In addition, since EDS is a local and semi-quantitative analysis technique, the measured composition may be influenced by the selected area and the local distribution of Cu-rich regions. Therefore, the difference between the nominal and measured Cu contents should be interpreted by considering both the progressive homogenization of Cu with increasing milling time and the local character of EDS analysis. In this respect, the increase in measured Cu content from NTC1 to NTC3 was found to be consistent with the improved Cu distribution observed in the elemental maps. Overall, it was observed that a fully homogeneous mixture could not be achieved in NTC1, whereas a more balanced elemental distribution was obtained in NTC2, and Cu atoms were largely integrated into the matrix in NTC3. However, under the NTC3 condition, agglomeration was observed in some powders due to increased deformation and cold welding. These results indicate that, although mechanical alloying time contributes to microstructural homogeneity, morphological integrity may be adversely affected beyond a certain point. When compared with the results reported by Cîrstea et al. (2024) and Aubakirova et al. (2025), the trends observed in the present study were found to be consistent with the literature, indicating that the selection of an appropriate mechanical alloying time is critical for achieving a homogeneous distribution of Cu.

Figure 6 shows the X-ray diffraction (XRD) patterns of samples NTC1, NTC2, and NTC3. In all samples, depending on the mechanical alloying time, the presence of phases such as NiTi, Ni_4Ti_3, Ni_3Ti, and Ti(Ni,Cu) was observed. The positions and intensities of the peaks clearly show the effect of alloying time on phase evolution. In addition, a progressive change in relative peak intensity and peak broadening was observed with increasing milling time, indicating a gradual evolution of the phase structure.

In the XRD pattern of the NTC1 sample, sharp peaks clearly belonging to the Ni_3Ti and Ni_4Ti_3 phases are seen together with the NiTi matrix. In addition, weak peaks belonging to the Ti(Ni,Cu) solid solution were detected. This result shows that in the early stage of alloying, diffusion between the NiTi matrix and Cu particles has not yet fully occurred. Similarly, Nasiri-Tabrizi and Fahami., (2013); reported that in short-time ($\leq$300 min) mechanical alloying the Ni_3Ti phase is dominant and longer time is needed for the alloy to become homogeneous.

In the NTC2 sample, it is observed that the intensities of the peaks belonging to the Ni_3Ti and Ni_4Ti_3 phases decreased, but they did not disappear completely. The NiTi and Ti(Ni,Cu) phases remained present. This situation shows that as the mechanical alloying time increases, Cu continues to partially diffuse into the NiTi lattice. The partial broadening in the peaks can be associated with increased lattice strain and defect density rather than a reduction in particle size. Compared with NTC1, the decrease in secondary-phase peak intensity and the more noticeable broadening of the diffraction peaks suggest that phase evolution proceeded further at 120 min. These results are consistent with the findings of Atiyah et al.,(2014); and Tanış et al., (2018); that Cu addition increases the diffusion rate and facilitates phase formation.

In the XRD pattern of the NTC3 sample, the NiTi phase became dominant, and the Ni_4Ti_3 and Ti(Ni,Cu) peaks were still present but relatively weaker compared to the dominant NiTi peaks. It is even seen that some weak peaks disappear after $2\theta \sim 50°$. This situation shows that with long-time mechanical alloying, Cu dissolves more homogeneously in the NiTi lattice and the system progresses into a nearly single-phase NiTiCu solid solution. The broadening of the peaks can again be attributed to the increase of residual stress and defect density in the crystal lattice as the mechanical alloying time increases. In semi-quantitative terms, the reduction in the visibility of secondary-phase peaks together with the dominance of the NiTi phase indicates that phase evolution became more pronounced at 180 min. These findings suggest that after long-time mechanical alloying, the NiTiCu solid solution becomes more stable and the phase transitions are largely advanced. Rezgoun et al., (2020); also stated similarly that after long-time mechanical milling, Ni_3Ti peaks disappeared and the NiTiCu phase became dominant.

The Ti(Ni,Cu) phase observed in all samples suggests that Cu atoms occupy the Ni sites in the NiTi lattice and behave as a solid solution within the NiTi structure without forming separate Cu phases. Rezgoun et al.,(2020), also argued in their study that Cu dissolves in Ti-rich regions and forms a stable Ti(Ni,Cu) solid solution. Overall, the XRD results indicate that increasing mechanical alloying time led to a gradual decrease in the relative intensity of secondary phases and a more pronounced peak broadening, pointing to continued phase evolution and increasing lattice distortion.

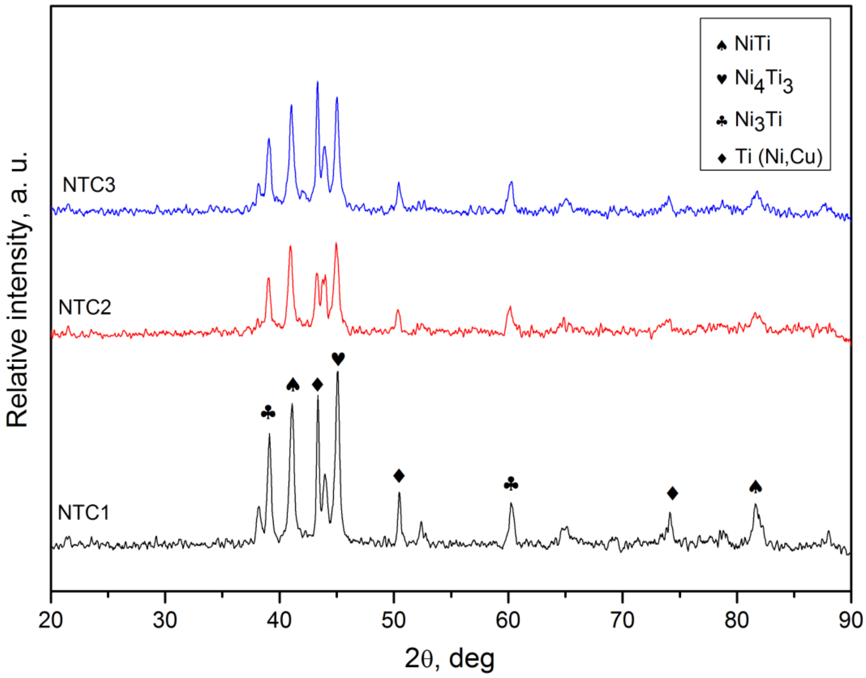

Figure 6 XRD patterns of NTC1, NTC2, and NTC3 samples

4. Conclusion

In this study, the $(NiTi)_{95}Cu_5$ wt% alloy was prepared by adding Cu to pre-alloyed NiTi powders and mechanically alloying them for 60, 120, and 180 min. The results showed that milling time had a direct effect on particle morphology, Cu distribution, and phase evolution. The average particle size increased from 50.9 µm for NTC1 to 51.6 µm for NTC2 and 61.1 µm for NTC3, while the standard deviation increased from 13.96 µm to 16.24 µm and 37.57 µm, indicating a broader particle size distribution with increasing milling time. These findings suggest that fragmentation and agglomeration occurred simultaneously during the mechanical alloying process.

EDS results showed that the measured Cu content increased from 2.7 wt% in NTC1 to 3.2 wt% in NTC2 and 5.1 wt% in NTC3, indicating that Cu distribution became more homogeneous with increasing milling time. XRD analyses revealed the presence of NiTi, Ti(Ni,Cu), Ni_4Ti_3, and Ni_3Ti phases in all samples. With increasing milling time, the relative intensities of Ni_3Ti and Ni_4Ti_3 peaks decreased, while the NiTi phase became more dominant, indicating progressive phase evolution toward a more stable NiTiCu-based structure.

The main scientific contribution of this study is the demonstration that even short-time mechanical alloying can produce significant microstructural changes in Cu-added pre-alloyed NiTi powders. The results show that increasing milling time improves elemental homogeneity and phase stability, but may also reduce morphological integrity due to increased deformation and agglomeration. Therefore, the controlled selection of short-time mechanical alloying conditions is important for balancing phase development, chemical homogeneity, and powder morphology. These findings provide a useful basis for the development of powder metallurgy-based NiTiCu shape memory alloys for low-temperature applications.

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Conflict of Interest Statement

The author declares that there is no conflict of interest.

Author Contribution Statement

The author declares a 100% contribution to the study.

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