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# Mechanochemical synthesis of niobium silicides: the effect of ball diameter and activation time on niobium and silicon mechanical activation

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**Abstract.** The paper investigated the regularities of mechanochemical formation of niobium silicides utilizing industrial waste (niobium turnings and silicon wafers) as feedstock. The influence of the milling ball diameter and the duration of mechanical activation on the mass fraction of formed niobium silicides was identified. X-ray phase analysis and scanning electron microscopy methods revealed that morphological changes are closely related to the formation of new phases such as $NbSi_2$, $Nb_5Si_3$, $Nb_3Si$. A high degree of comminution and particle deformation is required for successful mechanochemical synthesis, with activation time being a key factor. Maximum structural disordering and refinement are achieved in intermediate activation regimes (10–15 min). The use of larger balls (8 mm) intensifies deformation and disordering processes, leading to a sharper increase in microstrain and faster formation of nanoscale silicides. The total mass fraction of niobium silicides reached 46 wt. % when using 8 mm balls, compared to 38 wt. % when using 5 mm balls after 30 minutes of MA. The utilization of secondary waste as raw materials is suitable for niobium silicide production.

**Keywords:** niobium; silicon; niobium silicides; mechanical activation; morphology; mechanochemical synthesis; X-ray phase analysis; technological processing, industrial waste.

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# Механическая активация ниобия и кремния: роль диаметра шаров и времени в механохимическом синтезе силицидов ниобия

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**Аннотация.** Исследованы закономерности механохимического формирования силицидов ниобия с использованием техногенных отходов (стружка ниобия и пластины кремния) в качестве исходных материалов. Выявлены роль диаметра мелющих шаров и времени механической активации на изменения массовой доли образованных силицидов ниобия. Методами рентгенофазового анализа и растровой электронной микроскопии изучено и показано, что морфологические изменения тесно связаны с образованием таких новых фаз как $NbSi_2$, $Nb_5Si_3$, $Nb_3Si$. Высокая степень измельчения и деформации частиц является предпосылкой для успешного механохимического синтеза. Время механической активации является ключевым фактором в процессе механохимического синтеза силицидов ниобия. Максимальное разупорядочение и измельчение структуры,

достигается в промежуточных режимах активации (10…15 мин). Использование более крупных шаров (8 мм) интенсифицирует процессы деформации и разупорядочения, приводя к более резкому увеличению микроискажений и к более быстрому образованию наноразмерных силицидов. Массовая суммарная доля силицидов ниобия составила 46 мас. % при использовании шаров 8 мм против 38 мас. % при использовании шаров 5 мм после 30-минутной механической активации. Использование вторичных отходов в качестве исходных материалов пригодно для получения силицидов ниобия.

**Ключевые слова:** ниобий; кремний; силициды ниобия; механическая активация; морфология; механохимический синтез; рентгенофазовый анализ; технологическая обработка, техногенные отходы.

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## 1. Introduction

Transition-metal silicides, including niobium silicides, are promising for many applications owing to their high melting points, chemical stability in aggressive environments, high hardness, and thermal resilience. They exhibit excellent high-temperature performance, good oxidation resistance and retention of properties at elevated temperatures, as well as favorable electrical conductivity and chemical compatibility. These attributes enable their use in heat-resistant materials and as components of wear-prone high-temperature alloys, and in heat-resistant coatings [1–4]. Such properties also make them attractive for aerospace applications, heating elements, and protective coatings operating under extreme conditions, and they are considered potential substitutes for nickel-based alloys in heat-loaded components exposed to high temperatures [5]. Given their superior operational characteristics, research on the synthesis of these materials is of considerable scientific and practical importance. Mechanical activation (MA) is a widely used method, applied both for direct synthesis and as an effective preliminary treatment of materials [6–8].

Methods for synthesizing niobium silicides often require high temperatures and prolonged reaction times [9], which limits their applicability. In this context, mechanical activation (MA) for mechanochemical synthesis of niobium silicides appears promising. Single-phase $Nb_5Si_3$ has been successfully produced by high-energy impact loading and by self-propagating high-temperature synthesis following preliminary MA [10, 11], whereas formation of amorphous $Nb_{50}Si_{50}$ in a planetary mill required an extended milling time (150 h) [12]. A distinctive feature of mechanical activation is the simultaneous operation and interaction of many controllable process parameters. These parameters and their combined effects determine the evolution of the powder mixture and the subsequent phase-structural transformations of the material.

In the present work, technogenic waste – niobium metal turnings and silicon plates – were used as feedstock. The use of secondary resources addresses environmental and resource-saving goals, reduces raw-material costs, and minimizes waste, thereby promoting rational use of natural resources. International experience supports the effectiveness of using niobium and silicon wastes as raw-material sources [13, 21].

Under certain conditions, mechanochemical synthesis and mechanical alloying with formation of new phases occur during mechanical activation (MA). Interaction of powder particles with milling media (balls) and the drum walls produces heating, deformation, mixing, and comminution of the starting powder particles, the formation of layered agglomerates, an increase in interfacial area, welding, and the emergence of new phases and alloy particles [8, 14–17]. The feasibility of mechanochemical reactions and alloying depends on the characteristics of the mill, the selected mode, and the chosen operating parameters [18, 19]. Reaction progression in the contact zones is driven by the energy stored there, and the reaction yield is proportional to the total contact area between reagent particles, which depends on the number and diameter of the balls used [20]. The present study investigates the influence of MA time and ball diameter on the mechanochemical synthesis of niobium silicides.

The objective of the study is to determine the effect of milling ball diameter and MA time on the formation of niobium silicides during mechanochemical synthesis using technogenic waste as starting materials. To achieve this objective, the following tasks have been set: to perform mechanical activation of niobium and silicon while varying MA time and ball diameter; to investigate the morphology, phase composition, and structural state of the mechanically activated mixtures using X-ray phase analysis, X-ray structural analysis, and scanning electron microscopy.

## 2. Materials and Methods

### *2.1. Method for obtaining niobium silicides*

Niobium powder was produced by processing turnings of commercially pure niobium. The turnings were sheared with guillotine shears into platelets of approximately 5 × 5 × 2 mm, which were then milled in a ball mill ("Activator 2SL") for 10 min. Silicon powder was prepared from technical-grade silicon plates sized 100 × 100 × 5 mm. The plates were comminuted using a laboratory disintegrator (Desi 11).

A planetary mill "Activator 2SL" with water cooling was used for the mechanochemical synthesis of niobium silicides. MA conditions were as follows: drum/disc radius ratio 1.57, planetary radius 103 mm, drum volume (WC-Co, VK8) 200 mL. The disk and drum rotation speeds were 1000 and 1500 $min^{-1}$, respectively. WC-Co balls of 5 mm and 8 mm diameter were used as grinding media. A 100 g powder mixture of niobium and silicon, prepared to the stoichiometry of $Nb_5Si_3$, was activated for 5, 10, 15, and 30 minutes with a ball-to-powder mass ratio of 6.3 : 1.

### *2.2. Analytic methods*

The mechanically activated powders were analyzed by X-ray phase analysis (XRD-6000, Cu Kα radiation, PDF-4+ and POWDER CELL 2.4 databases) and by scanning electron microscopy (Quanta 200 3D). Crystallite sizes were estimated using the Scherrer equation. Rietveld refinement was applied to the X-ray diffraction patterns to separate contributions from crystallite size and microstrain and to assess their uncertainties; this refinement accounted for both instrumental broadening (via instrument parameters) and physical broadening (related to crystallite size and microstrain). Statistical uncertainties for the refined parameters (including crystallite size and microstrain) ranged from 3.4 to 6.2 %. Specific surface area was measured by nitrogen thermal desorption using the four-point BET method on a SORBI-M instrument, with nitrogen as the adsorbate. Prior to measurement, powders were degassed by thermal treatment for 1 hour at 200 °C.

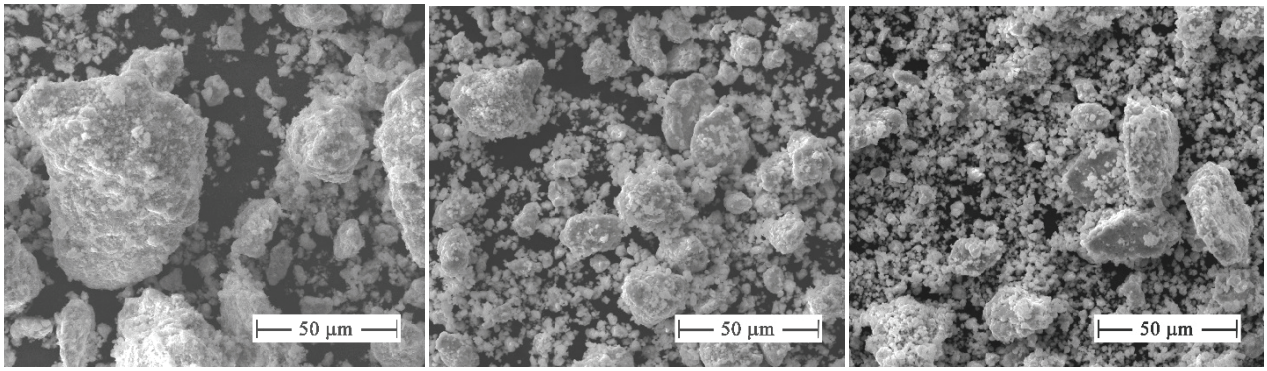
## 3. Results and Discussion

Figure 1 shows a scanning electron microscope (SEM) image of a niobium-silicon powder mixture obtained after 5 minutes of mechanical activation (Fig. 1*a*). The image demonstrates particle morphology indicating the initial stages of intense particle crushing and agglomeration. The dominant structural element is large agglomerates, reaching sizes comparable to 50 μm. These agglomerates have a non-uniform, lumpy surface, indicating their formation from smaller particles under the influence of shock-shear loads. The surface of the agglomerates bears traces of intense mechanical action: roughness and the presence of small particles adhering to larger ones. A significant number of small particles are visible between the large agglomerates. Some of these small particles appear more angular, while others are smoothed, which may reflect different stages of mechanical processing. During this time interval (5 min MA) the destruction of the original particles occurs and the simultaneous formation of new, smaller ones, which begin to agglomerate, sticking to each other.

A SEM-image of a niobium-silicon powder mixture after 15 min of MA (Fig. 1*b*) demonstrates differences from the morphology observed after 5 minutes of MA, where large agglomerates with a non-uniform surface predominated. After 15 min, a finer grinding was observed. The number of small, freely distributed particles increased. The main agglomerates appear smaller and consist of even smaller components. The total volume of the powder mixture now consists of a larger number of small particles. This indicates that the grinding process, crushing both the original particles and the previously formed agglomerates, continued during the additional 10 minutes of MA (from 5 to 15 min). While after 5 min of MA, the agglomerates had a more "sculpted" shape with clear signs of clumping, after 15 min they appear "loose", consisting of smaller particles. The increased degree of dispersion and the increase in the interphase surface of the agglomerates create favorable conditions for the occurrence of mechanochemical reactions, and promote diffusion processes and the formation of silicide phases.

A comparative analysis (5 min MA *vs*. 15 min MA) shows that after 15 minutes of MA, a decrease in the average particle size and an increase in the proportion of fine particles were observed compared to 5 minutes of MA. Increased crushing and agglomeration processes created the preconditions for the formation of layered agglomerates and an increase in the interfacial surface area, creating conditions for the mechanochemical synthesis of niobium silicides. These changes indicate that increasing the MA time prepares the material for the formation of the target phases.

Figure 1*c* shows the SEM-image of a niobium-silicon powder mixture after 30 min of MA, where




*(a)* *(b)* *(c)*

**Fig. 1.** SEM-images of the silicon-niobium powder mixture after: *a* – 5 min MA; *b* – 15 min MA; *c* – 30 min MA using 5 mm balls

further changes in morphology were visible and the finest grinding was observed. Most of the particles were smaller than in previous stages. Fine material was formed, with micron and submicron particles predominating. This indicates an intensive crushing process that continued throughout the entire MA period. While large agglomerates predominated after 5 minutes of MA, and became looser after 15 min, after 30 min large agglomerates were virtually absent. Clusters of very small particles were observed, which began to form new, smaller and looser aggregates that lacked the distinct structure of the primary, large agglomerates. This structure facilitates more efficient mechanochemical reactions. A morphology characterized by strong comminution and a high degree of particle deformation often correlates with the onset of amorphous or nanostructured phase formation [8, 11, 14, 19]. The analysis of the powder mixture after 30 min of MA demonstrated a transition to a highly dispersed state with a predominance of very small particles.

Thus, analyzing the samples after 5, 15, and 30 min of mechanical activation, we see that after 5 min of mechanical activation, large agglomerates with pronounced traces of impact-shear action predominated. Small particles were relatively few. After 15 minutes of mechanical activation, the agglomerate size decreased, the proportion of small particles increased, with looser aggregates formed. The grinding process continued actively. After 30 minutes of mechanical activation, a high degree of dispersion was observed. Very small particles predominated, with virtually no large agglomerates. An increase in the specific surface area was observed, creating optimal conditions for mechanochemical synthesis.

Thus, the morphological changes in the niobium-silicon powder mixture observed with increasing mechanical activation time indicate progressive degradation of the starting materials and the preparation of the powder structure for the formation of new compounds.

Figure 2 shows the changes in the specific surface area of the powder mixture depending on the MA time using grinding balls with diameters of 5 and 8 mm. The specific surface area for the 5 mm diameter (square) initially increased sharply from the initial state (presumably up to 5 min of MA, where it is about 2.4 $m^2 \cdot g^{-1}$), then decreased by 15 min (about 1.1 $m^2 \cdot g^{-1}$), and again increases slightly by 30 min (about 1.4 $m^2 \cdot g^{-1}$). The specific surface area for the 8 mm diameter (circle) also initially decreased from the initial value (about 1.9 $m^2 \cdot g^{-1}$ at 5 min of MA), reached a minimum at 15 minutes (about 1.3 $m^2 \cdot g^{-1}$), and then increased slightly by 30 minutes (about 1.4 $m^2 \cdot g^{-1}$). The analysis of the effect of grinding ball size (5 mm *vs*. 8 mm) shows that using 8 mm balls resulted in a lower specific surface area at early stages (5 and 10 min of MA), but by 15 and

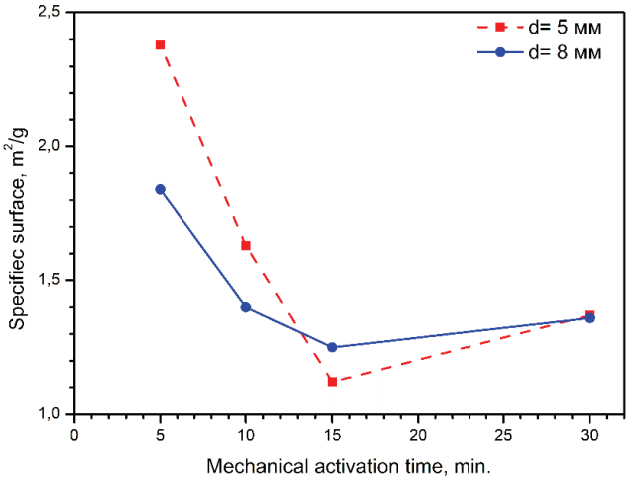
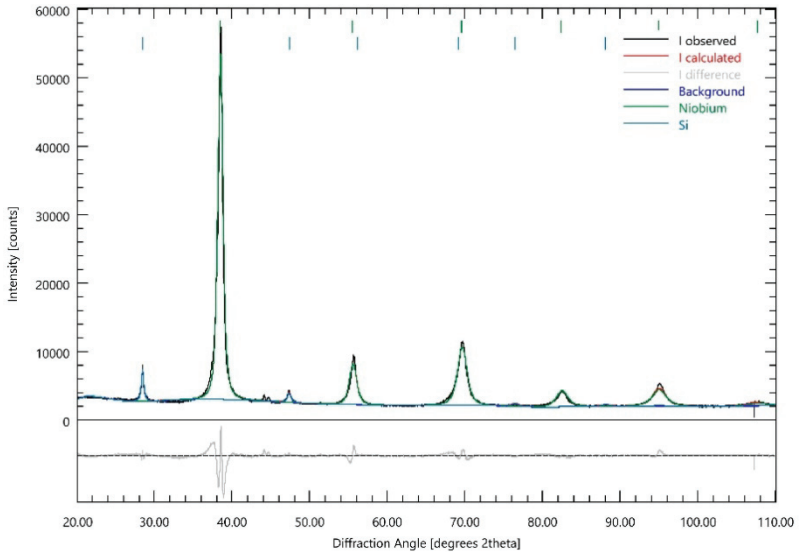
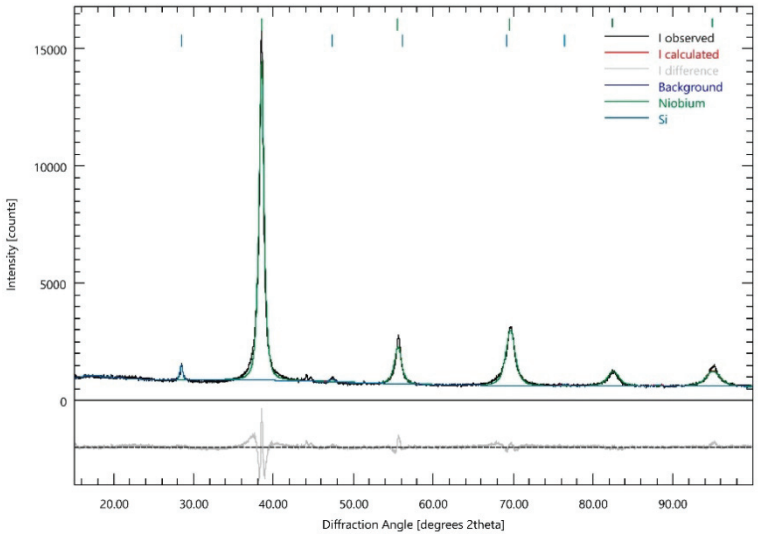


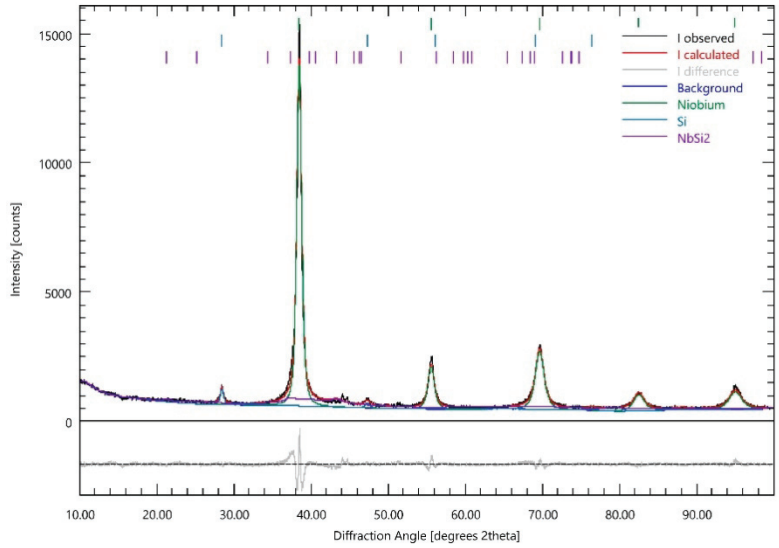

**Fig. 2.** Specific surface area as a function of mechanical activation time using 5 mm diameter balls (■) and 8 mm diameter balls (●)




*(a)*




*(b)*



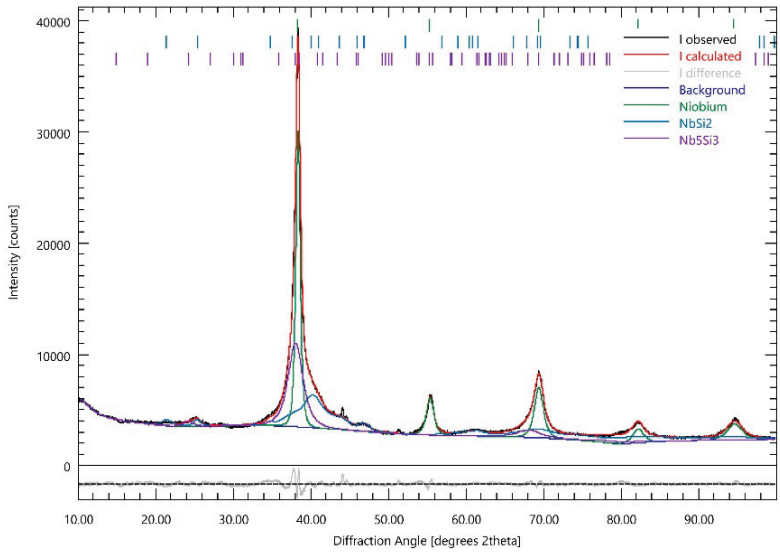

*(c)*

**Fig. 3.** Powder mixture diffractogram fragments: *a* – initial mixture, after 5 min MA, obtained using ball diameters: *b* – 5 mm; *c* – 8 mm. Nb (#01-077-3017), Si (#00-026-1481), NbSi2 (#01-0746870)

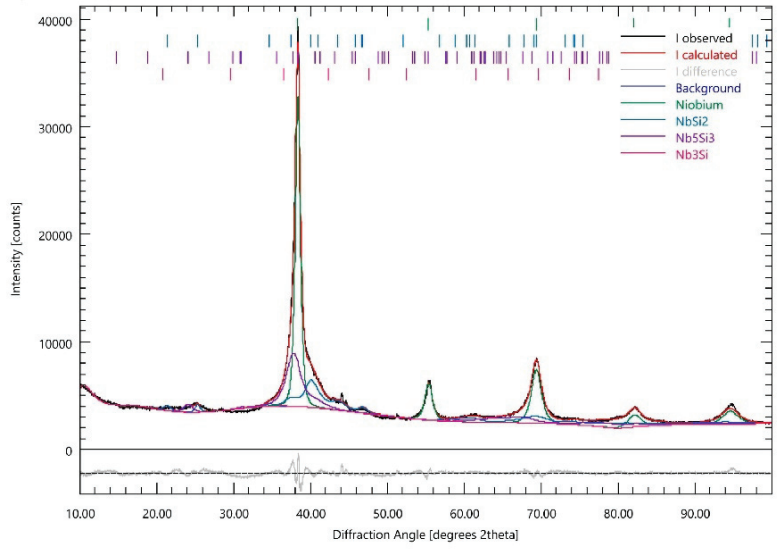
30 min of MA, the values became similar. This is consistent with the assumption that 8 mm balls provide higher MA intensity but lead to more rapid agglomeration and powder compaction, which is reflected in the specific surface area curve.

Figure 3 shows fragments of the diffraction patterns after 5 min of MA for ball diameters of 5 (Fig. 3*a*) and 8 mm (Fig. 3*b*). The diffraction patterns in Fig. 3*a* show intense peaks corresponding to Nb and Si, while Figure 3b also shows the initial Nb and Si, but with noticeable peaks related to the $NbSi_2$ phase. This indicates that using larger balls (8 mm) at the same MA time (5 min) results in a slightly more active reaction onset.

Figure 4 shows fragments of the diffraction patterns after 30 min of MA for balls with diameters of 5 and 8 mm. When using 5 mm balls, the appearance of the diffraction pattern differed significantly from the case of 5 min of MA. Now there are pronounced peaks corresponding to niobium silicides: $NbSi_2$ (marked as "1") and $Nb_5Si_3$ ("2"). The peaks of Nb and Si were also preserved, but their intensity decreased. When using 8 mm balls (Fig. 4*b*), a fragment of the diffraction pattern also shows the presence of silicides, but here, in addition to $NbSi_2$ ("1") and $Nb_5Si_3$ ("2"), peaks of $Nb_3Si$ ("3") were observed. The intensity of the niobium silicide peaks, especially $NbSi_2$ and $Nb_5Si_3$, was significantly higher here than for 5 mm balls at the same MA time (30 min). The intensity of the initial niobium was lower than for 5 mm balls.




*(a)*




*(b)*

**Fig. 4.** Powder mixture diffractogram fragments after 30 min MA, obtained using ball diameters: *a* – 5 mm; *b* – 8 mm

The SEM-image after 30 min of MA (Fig. 1*c*) shows a high degree of grinding and dispersion, which is completely consistent with the diffraction data. It is precisely at this fine grinding that the intensive formation of new phases (niobium silicides) occurs. The higher intensity of the silicide peaks for 8 mm balls at 30 min of MA, compared to 5 mm balls, may be due to more effective mechanical alloying and/or the higher temperature generated during impacts with the larger balls, which facilitates the reactions.

Figure 5 shows the dependence of the change in the mass fraction of the initial components and the formed silicides on the MA time, as determined by X-ray diffraction data. The graph provides a quantitative assessment of the phase composition as a function of MA time and sphere size.

The proportion of initial niobium (curve *1*, squares) decreased with MA time, especially noticeably after 15 minutes of MA. This confirms that Nb actively participates in silicide formation reactions. The proportion of initial silicon (curve *2*, circles) also decreased, but less dramatically than the proportion of Nb. This is due to the fact that it was used in smaller quantities (based on the stoichiometry of $Nb_5Si_3$). The proportion of mechanochemically formed niobium silicides (curve *3*, triangles) increased with MA time.

The analysis of the influence of the ball size (5 mm *vs*. 8 mm) according to XRD shows that after 5 min of MA: when using 8 mm balls (Fig. 3*b*, and curve *3* in Fig. 5*b*) a higher proportion of silicides was observed than with 5 mm balls (Fig. 3*a*, and curve *3* in Fig. 5*a*). This confirms that larger balls contribute to a faster reaction start. After 30 min of MA for 5 mm balls (Fig. 4*a*, Fig. 5*a*) the proportion of silicides reached approximately 38 % and for 8 mm balls (Fig. 4*b*, Fig. 5*b*) the proportion of silicides was about 46 %, and a greater variety of phases was present ($Nb_2Si$, $Nb_5Si_3$, $Nb_3Si$). Figure 5*b*, curve *3*, also shows an increase in the proportion of silicides, comparable to that in Fig. 5*a*. After 30 min of the MA process for 5 mm balls, the predominant silicide was $NbSi_2$ (30 wt. %), with Nb5Si3 accounting for 8 wt%. For 8 mm balls, with the same MA time, the $NbSi_2$ content was also 30 wt. %, $Nb_5Si_3$ – 1 wt. %, and $Nb_3Si$ (15 wt. %) was added.

Thus, using 8 mm balls accelerates the silicide formation process and can facilitate the formation of a more diverse set of phases.

Figure 6 shows the dependences of changes in coherent scattering areas (CSA) on the MA time. CSA (crystallite size) is the average size of regions within the material where the crystalline structure remains continuous. During MA, the crystals were crushed, and the CSA decreased. When using 5 mm balls, (Fig. 6*a*) the CSA size for Nb (*1*) remained relatively stable within 10–20 nm, with a noticeable decrease at 15 min of MA. The CSA size for Si (*2*) initially increased slightly (up to 10 min of MA) and then decreased. The CSA size for niobium silicide $Nb_5Si_3$ (*3*), which began to form, initially decreases (up to 10 min) and then increased. This may be due to the initial formation of nanoparticles, which then began to agglomerate or enlarge. When using 8 mm balls, (Fig. 6*b*) The CSR size for Nb (*1*) also decreased with MA time, remaining within 10-20 nm.

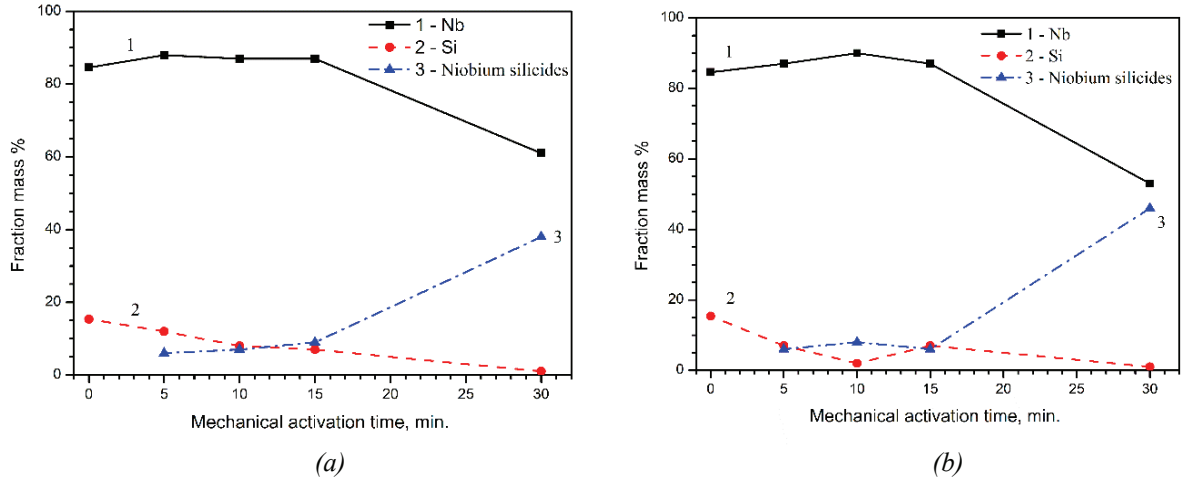
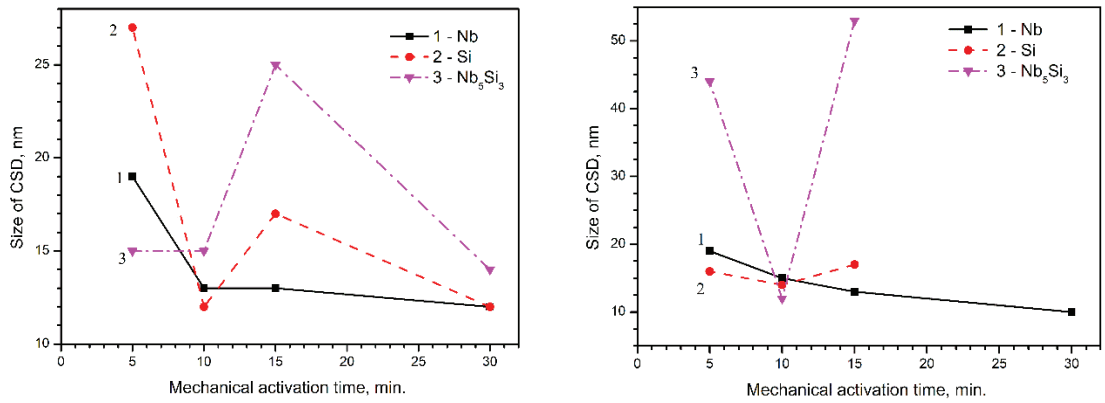



**Fig. 5.** XRD-based mass fraction changes of initial components and mechanochemically formed silicides, using ball diameters: *a* – 5 mm; *b* – 8 mm




*(a)* *(b)*

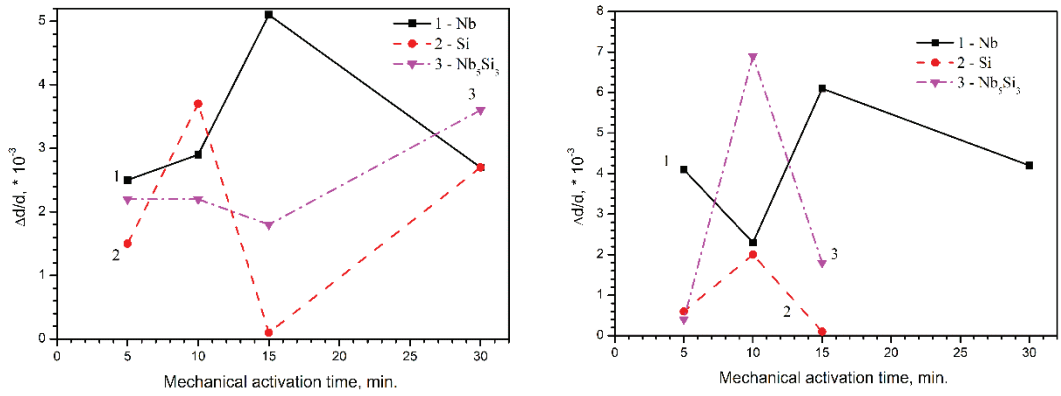
**Fig. 6.** Evolution of crystallite size (coherent scattering domain size) for initial components and mechanochemically formed niobium silicides as a function of mechanical activation time, with ball diameters: *a* – 5 mm; *b* – 8 mm

The CSR size for Si (*2*) remained relatively low and stable. The CSR size for niobium silicide $Nb_5Si_3$ (*3*) behaved similarly to that for 5 mm spheres, with a sharp increase after 15 min of MA and then a decrease. However, after 5 minutes of MA, the CSR of the silicide was already significantly smaller than that for 5 mm spheres, which may indicate more rapid formation of nanoscale regions.

The decrease in the CSR for the initial components during MA indicates their refinement and disordering of the crystalline structure. This is consistent with SEM-images, which showed progressive particle refinement. The increase and subsequent decrease in the CSR for silicides may reflect the process of their formation and refinement of already formed phases.

Figure 7 shows the dependence of changes in crystal lattice microdistortions ($\Delta d/d$) on the MA time. Microdistortions (or deformations) of the crystal lattice are a violation of the ideal periodicity of the crystal structure. MA causes deformations and the accumulation of vacancies and dislocations, leading to an increase in microdistortions. For 5 mm spheres, microdistortions for Nb (*1*) and Si (*2*) initially increased (up to 15 min of MA), reaching a maximum, and then began to decrease. Microdistortions for $Nb_5Si_3$ (*3*) tended to increase over time, reaching their maximum at 15 min of MA and then decreasing. This may be due to relaxation processes and some annealing with increasing temperature.

For 8 mm spheres, microdistortions for Nb (*1*) initially increased, reaching a maximum at 10 min of MA, and then decreased, while for Si (*2*) they remained relatively low and stable. For 8 mm balls, the peak microdistortions for Nb and $Nb_5Si_3$ were reached earlier (around 10 min) and were more pronounced. This indicates a more intense and rapid accumulation of defects when using larger balls. The peak microdistortions for $Nb_5Si_3$ for 8 mm balls after 10 min of MA were very high (more than $7 \cdot 10^{-3}$), indicating significant disordering of its crystal lattice. This may be due to the more active formation of nanoscale or amorphous regions under more intense mechanical activation.




*(a)* *(b)*

**Fig. 7.** Evolution of lattice microstrain in initial components (*1* – niobium, *2* – silicon) and mechanochemically formed niobium silicides (*3* – $Nb_5Si_3$) as a function of mechanical activation time, with ball diameters: *a* – 5 mm; *b* – 8 mm

An increase in microdistortions with increasing MA time indicates the accumulation of defects in the crystal lattice of the starting components and the forming silicides. A decrease in the coherent scattering area and an increase in microdistortions occur in parallel, reflecting the intensity of deformation processes. High microdistortion values and low coherent scattering area values (especially at 15 min of MA) correlate with the high degree of dispersion observed in SEM-images.

Thus, the use of larger grinding balls (8 mm), all other things being equal, accelerates the MA and synthesis process, leading to more rapid formation of silicides and the formation of a more complete set of silicide phases.

## 4. Conclusion

It was found that with increasing MA time, particle refinement occurred, their specific surface area increased, and, consequently, the proportion of formed silicide phases increased. According to X-ray diffraction data, maximum disordering and structure refinement were achieved in intermediate activation modes (10–15 min), which correlates with the specific surface area and morphology data. Using larger balls (8 mm) intensified the deformation and disordering processes, leading to a sharper increase in microdistortions and faster formation of nanoscale silicides. The total mass fraction of niobium silicides was 46 % when using 8 mm balls versus 38 % when using 5 mm balls after 30 minutes of mechanical activation.

The use of secondary waste as starting materials is suitable for producing niobium silicides. With increasing MA time, particle refinement occurred, their specific surface area increased, and, consequently, the proportion of formed silicide phases increased. Mechanical activation leads to crystallite refinement (reduction in CSR) and defect accumulation (increased microdistortions) in the starting components and the resulting silicides. According to X-ray diffraction data, maximum disordering and structure refinement were achieved in intermediate activation modes (10–15 min), which correlates with the specific surface area and morphology data. The use of larger balls (8 mm) intensified the deformation and disordering processes, leading to a sharper increase in microdistortions and faster formation of nanoscale silicides. The total mass fraction of niobium silicides was 46 wt. % when using 8 mm balls versus 38 wt. % when using 5 mm balls after 30 minutes of mechanical activation. The use of secondary waste as starting materials is suitable for producing niobium silicides.

## 5. Funding

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## 7. Conflict of interests

The authors declare no conflict of interest.

## Конфликт интересов

Авторы заявляют об отсутствии конфликта интересов.

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