Article

Exploring the Impact of Pre-Mechanical Activation of Nickel Powder on the Structure of Deposited Metal: A Deep Neural Network Perspective

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Abstract: This study explores the potential application of the mechanical activation (MA) of nickel powder for incorporation into the composition of powder wire blends for the deposition of wear-resistant coatings. Nickel powder of PNE-1 grade was processed in a vibrational mill for various durations (4 to 16 min) with different combinations of grinding media. The influence of MA parameters on the bulk density and apparent particle size of nickel powder was investigated. The greatest effect was observed at the maximum processing time of 16 min, where electron microscopy revealed significant deformation and an increase in discoid particles, leading to enhanced energy accumulation. Nickel powder processed with a combination of 6 balls that are 20 mm in diameter and 8 balls that are 10 mm in diameter showed significant changes, though no major alteration in chemical composition was noted. XRMA indicated that the powder’s surface was partially covered with oxides, with a composition of 96.8–98.4% Ni and 0.8–1.7% O₂. Additionally, the effect of nickel powders after the treatment on the structure of deposited metal was determined, demonstrating alterations in the morphology and a slight increase in hardness. Furthermore, a convolutional neural network (CNN)-based approach was proposed to discern fragments within images depicting surface microstructures, both with and without MA.

Keywords: mechano-activation; nickel; surfacing; powder wire; microstructure; hardness

1. Introduction

Mechanical activation (MA) refers to the activation of solid phases through mechanical action [1]. The MA process involves mechanical impact on solid bodies resulting from impulsive shocks. Pre-mechanical activation of powders for preparing raw materials for subsequent technological operations finds broad applications in science and engineering [2–5]. Such powder treatment involves energy accumulation in crystals in the form of defects or other changes in the solid substance, which enables a reduction in the activation energy of subsequent chemical transformations [6]. Typically, MA is not a final operation but is used as a preparatory step for powders or powder mixtures before conducting subsequent technological processes. Powders are subjected to MA before self-propagating high-temperature synthesis, cold or hot pressing, additive manufacturing, thermal spraying, and in the manufacture of welding materials.
There is extensive experience in applying MA for the mechanical alloying of powders, which are subsequently used either as standalone filler materials in plasma powder deposition [7], or as additional metallurgical additives or fluxes in flux-cored arc welding [8]. Mechanically alloyed powders are also incorporated into the composition of coated electrodes [9,10], fluxes [9,11], or powder wires [9,12]. In this context, MA is applied to mechanically alloyed powders of metal (babbitt, iron, or nickel) with nanoparticles (carbides, oxides, carbon nanotubes, etc.). Mechanical alloying is achieved by co-processing metal powders with nanoparticles, resulting in composite granules consisting of two phases. This approach facilitates the transportation of nanoparticles into the weld pool, shields nanoparticles from high-temperature effects, and modifies the structure of deposited metal coatings and welds.

The pre-mechanical activation of individual powders, particularly nickel powder, known for its ductility and energy accumulation capability, has been shown to positively influence the formation of the structure and properties of deposited metals. The utilization of nickel in powder wires is actively discussed in the scientific community due to its significant impact on enhancing the mechanical properties and structural integrity of the resulting materials.

Several studies have delved into the various aspects of mechanical activation and synthesis involving nickel and its compounds. Filimonov et al. [13] presented a comprehensive mathematical modeling of high-temperature synthesis in a Ni+Al powder system under linear heating conditions. Their work compared numerical modeling with experimental data for mechanically activated (MA) powder mixtures and proposed a criterion for determining the characteristic ignition temperature in forced ignition scenarios. The dependencies of ignition temperature, induction time, and conversion degree on effective activation energy were established, offering a novel method for determining the effective activation energy of synthesis based on the proposed criterion.

Exploring the mechano-chemical synthesis route, Nazemi et al. [14] investigated the production of nanostructured nickel aluminate spinel powder from NiO/Al₂O₃ spent catalysts. Their characterization studies using XRD, SEM, TEM, DTA, and nitrogen adsorption revealed spinel formation after 60 h of milling without heat treatment. The mechanical activation influenced the heat treatment temperature, demonstrating that 15 h of milling followed by treatment at 1100 °C was sufficient for spinel production. This direct mechanical milling approach yielded a higher surface area and smaller crystallite size compared to the heat-treated product.

Savic et al. [15] described the synthesis of nickel manganite powder through the calcination of a stoichiometric mixture of manganese and nickel oxide, followed by mechanical activation in a high-energy planetary ball mill. This method achieved a pure NiMn₂O₄ phase, which was then pressed into disc-shaped pellets and sintered. The study employed scanning electron microscopy and X-ray powder diffraction to track changes in particle morphology and structural characteristics, respectively. AC impedance spectroscopy on sintered samples revealed that mechanical activation enhances transport processes and decreases average crystallite size, though prolonged activation times could lead to aggregate formation, defects, and increased lattice microstrains.

Additionally, Zhao et al. [16] focused on synthesizing a NiAl/TiC₀.₉₅ composite via reactive spark plasma sintering of MA elemental powders. The microstructure and properties of both activated powders and sintered samples were evaluated. The sintering process resulted in the elimination of certain phases while retaining others, with nanoindentation tests showing a hardness of 12.2 ± 0.1 GPa and an elastic modulus of 25.0 ± 0.5 GPa, indicative of the material’s enhanced mechanical properties.

In a different application, Kormaz et al. [17] proposed a hybrid method for mechanical activation in cement production to reduce duration and energy consumption. By integrating a roller press and a hammer grinder before a ball mill, their method demonstrated improved reactivity and reduced energy consumption. Among the tested hybrid grinding
methods, the integration of the roller press and ball mill emerged as the superior mechanical activation approach.

Table 1 summarizes the focus and limitations of these studies. This table highlights the range of applications investigated, from the use of MA in enhancing material properties in welding to the theoretical modeling of synthesis processes. It also identifies the limitations of these studies, such as their specific focus on other materials or theoretical aspects rather than practical applications of MA-treated nickel powders.

Table 1. Summary of Related Work on Mechanical Activation and Nickel Powders.

<table>
<thead>
<tr>
<th>Reference</th>
<th>Focus</th>
<th>Limitations</th>
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<tbody>
<tr>
<td>Kobernik et al., 2013 [7]</td>
<td>Application of MA for mechanical alloying of powders used in plasma powder deposition.</td>
<td>Limited to antifriction materials and does not explore nickel specifically.</td>
</tr>
<tr>
<td>Sokolov et al., 2009 [9]</td>
<td>Incorporation of mechanically alloyed powders into coated electrodes and fluxes.</td>
<td>Broad application but lacks specific emphasis on pre-mechanical activation of nickel powder.</td>
</tr>
<tr>
<td>Nazemi et al., 2012 [14]</td>
<td>Mechano-chemical synthesis of nanostructured nickel aluminate spinel powder.</td>
<td>Specific to nanostructured spinel, not broadly applicable to all nickel powders.</td>
</tr>
<tr>
<td>Zhao et al., 2023 [16]</td>
<td>Synthesis of NiAl/TiC\textsubscript{0.95} composite via reactive spark plasma sintering of MA powders.</td>
<td>Focuses on specific composite material, not general use of MA nickel powders.</td>
</tr>
<tr>
<td>Kormaz et al., 2022 [17]</td>
<td>Hybrid mechanical activation method in cement production to reduce energy consumption.</td>
<td>Application limited to cement production, not applicable to metal powders.</td>
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Despite significant advancements in the development of wear-resistant coatings, there remains a gap in understanding the effects of mechanical activation (MA) on nickel powder when incorporated into powder wire blends for deposition. Previous studies have not sufficiently explored the impact of MA parameters on the bulk density and particle size of nickel powder, nor their subsequent influence on the microstructure and properties of the deposited metal. This study aims to address these gaps by investigating the potential application of MA for nickel powder of PNE-1 grade, processed in a vibrational mill for varying durations and with different grinding media combinations. We examine how these MA parameters affect the bulk density and particle size of the powder and, subsequently, the structure and hardness of the deposited metal. Additionally, we propose a convolutional neural network (CNN)-based approach to analyze surface microstructure images, aiming to provide a comprehensive understanding of the changes induced by MA treatment.

2. Materials and Methods

The influence of MA was investigated on PNE-1 [18] grade nickel powders (Figure 1) according to GOST 9722 [19], with a purity of 99.5%, a bulk density of no more than 3.4 g/cm\textsuperscript{3}, and a size of less than 71 µm.

The study was conducted in two stages:

1. First Stage: The specified powder was subjected to MA followed by studying changes in bulk density and apparent particle size. X-ray spectroscopic microanalysis (XRMA) was also conducted. A total of 10 studies were performed at this stage.
2. Second Stage: The influence of processed powders on the structure and properties of deposited metal was investigated by introducing powders into the molten metal of the welding pool. This stage included 15 studies.

![Figure 1. Appearance of the nickel powder used.](image)

Powder processing was conducted using a ball vibrational mill, model ML-1, featuring a container made of 01X18H10T steel. The mill employed grinding media consisting of 20 mm and 10 mm diameter steel balls, also made from the same steel. The ML-1 vibrational mill is designed for intermittent dry fine grinding and homogenization of small samples (ranging from 2 to 50 g) with an initial particle size of 3 mm and a final size of 0.06 mm. It is suitable for preparing dispersions of various materials, including glass, ceramics, soils, food products, plant materials, and pharmaceutical components. The ML-1 is used for sample preparation in physical-chemical, spectral, and other analytical studies.

The mill operates with vertical vibrations generated by an electric motor and an eccentric mechanism. These vibrations are transmitted through the processed material to the grinding balls. The mill is equipped with two sizes of grinding cups, optimized for different sample quantities. The grinding cups are made of titanium, while the grinding balls are steel with a titanium coating. This setup ensures rapid grinding through impact and friction, with minimal loss of material. The working chamber is hermetically sealed with a cover to ensure high fineness of the final grind. The grinding accessories are easy to clean, and the grinding cups are quickly and securely fixed in place.

Milling was conducted at a vibration frequency of 1490 min\(^{-1}\). Various quantities and combinations of grinding media were tested: in the first case, 7 balls of 20 mm diameter; in the second case, 6 balls of 20 mm diameter; and in the third case, 6 balls of 20 mm diameter combined with 8 balls of 10 mm diameter. Throughout the mechanical activation process, the mass ratio of powder to balls was consistently maintained at 1:10. The processing time varied from 4 to 16 min, with intervals of 4 min.

Bulk density was determined according to GOST 19440 [20] using a funnel method. A funnel with a diameter of 2.5 ± 0.05 mm and a cylindrical container with a capacity of
25 ± 0.05 cm³ and an inner diameter of 30 ± 1 mm were used. The mass of the powders was measured using VM213 (Torunit, China) scales with a weighing accuracy of 0.001 g. Measurements were conducted on three samples.

Bulk density was calculated using the equation:

$$\rho = \frac{m}{V}$$  \hspace{1cm} (1)

where $\rho$ is the bulk density of the powder in g/cm³, $m$ is the mass of the powder in grams, and $V$ is the volume of the powder in cm³ ($V = 25$ cm³).

The fractional composition of powders was determined by sieving the powder material through a set of sieves with sequentially decreasing hole sizes: 300 µm—200 µm—100 µm—88 µm—75 µm. Subsequently, the residue on each sieve was weighed with an accuracy of 0.001 g.

Based on the obtained fractional composition of powders, the apparent particle size of the powders was determined. The apparent particle size was calculated as the sum of the products of the relative quantity of powder remaining on a specific sieve and the size of that sieve. Additionally, the powder sieved through the last sieve was multiplied by half the average value of the nickel powder, according to GOST 19440 for this powder. Thus, the apparent particle size of the powder was determined using the formula:

$$d = \left( \sum \frac{m(x)}{M} \cdot D(x) + \frac{m(z)}{M} \cdot \frac{D(z)}{2} \right)$$  \hspace{1cm} (2)

where:

- $d$—average particle size of the powder (µm),
- $m(x)$—mass of the powder (g) remaining on the sieve of size $x$ (µm),
- $M$—total mass of the powder (g),
- $D(x)$—hole size on the sieve $x$ (µm),
- $m(z)$—mass of the powder sieved through the last sieve of size $z$ (g),
- $D(z)$—hole size on the last sieve $z$ (µm).

Microstructural analysis and chemical analysis of powders were conducted using XRMA on a Phenom Pharos microscope (Thermo Fisher Scientific Inc., Waltham, MA, USA).

At the second stage, the influence of introducing processed powders into the molten weld pool on the structure and hardness of the deposited metal was investigated. To introduce the processed powders into the molten weld pool, grooves with a width of 1.3 ± 0.05 mm and a depth of 2.0 ± 0.05 mm were machined on plates made of St3sp steel (according to GOST 380 [21]) with dimensions of 150 mm × 150 mm × 9 mm. Subsequently, the grooves were filled with powder through a funnel with a hole diameter of 2.5 mm until the powder filled the grooves. Then, the surface of the powder was leveled using a non-magnetic ruler without applying pressure to the filled powder. After powder filling, remelting of the filled grooves was carried out by arc welding under flux according to the experimentally selected mode presented in Table 2. An experimental powder wire (4.8 wt% C, 4.0 wt% Cr, 3.3 wt% Nb, 1.0 wt% B, 7.7 wt% Si) was used as the electrode wire. Additionally, ceramic neutral flux of 860 grade from Lincoln Electric (Euclid, OH, USA) was used.

<table>
<thead>
<tr>
<th>$I_{arc}$, A</th>
<th>$U_{arc}$, V</th>
<th>$V_{arc}$, m/h</th>
<th>$V_{wire}$, m/min</th>
</tr>
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<tr>
<td>200–210</td>
<td>37–38</td>
<td>16</td>
<td>2.2</td>
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After remelting the powders, specimens were prepared from the obtained samples for further microstructural analysis [22]. Before conducting microstructural analysis, the specimens were ground, polished, and then etched in a 4% nitric acid solution in alcohol.
Microstructural analysis was performed using optical microscopy on a ZEISS Axiovert 200MAT/200 M MAT microscope (Carl Zeiss Light Microscopy, Göttingen, Germany). Hardness was measured on the HRC scale using a TK-2M (Ivanovo plant of testing devices, Ivanovo, Russia) hardness tester with a cone indentation under a load of 1500 N.

3. Results

3.1. First Stage

The studies conducted in the first stage allowed us to establish that during the processing of nickel powder with balls for 4 min, the change in bulk density is within the measurement error (Figure 2). Both Mode 1 and its variation exhibit a minimal change in bulk density, indicating that the processing conditions for Mode 1 are stable and produce consistent results. Mode 2 and its variation might show a slightly higher or lower change in bulk density, pointing to some sensitivity to parameter variations. However, the changes still fall within the measurement error. Like Modes 1 and 2, Mode 3 and its variation maintain bulk density changes within the measurement error, implying that even with variations, the process does not significantly alter the bulk density.

![Figure 2. Change in bulk density of nickel powder depending on processing time for different container loads: (1) 7 balls with a diameter of 20 mm, (2) 6 balls with a diameter of 20 mm, (3) 6 balls with a diameter of 20 mm and 8 balls with a diameter of 10 mm.](image)

It is worth noting that for the container loaded with 7 balls with a diameter of 20 mm, this trend is observed up to 16 min of processing. However, when analyzing the apparent particle size (Figure 3), a dependency indicating a decrease in the apparent particle size with increasing processing time for loading with seven 20 mm diameter balls is traced. For processing with 7 balls of 20 mm diameter, at 4 min, the particle size is larger compared to the subsequent stages. By 8 min, the particle size shows a noticeable decrease. At 12 min, the reduction in particle size continues, indicating that the longer processing time facilitates breaking down the particles further. By 16 min, the particle size reaches a lower value, suggesting that the extended processing time consistently reduces the particle size. For 6 balls of 20 mm diameter, the particle size also decreases with increased processing time but may not reduce as significantly as with 7 balls. For 6 balls of 10 mm diameter, similar trends of particle size reduction are observed, though the final sizes differ slightly. For 8 balls of 10 mm diameter, there is a consistent decrease in particle size, comparable to the 7 balls of 20 mm diameter configuration.
Figure 3. Change in apparent particle size of nickel powder depending on processing time for different container loads: 7 balls with a diameter of 20 mm, 6 balls with a diameter of 20 mm, 6 balls with a diameter of 20 mm and 8 balls with a diameter of 10 mm.

Analysis of the obtained particles using SEM revealed that with an increase in MA time, there is a slight change in the shape of the processed powders and the contact area of the original particles. Dimples are observed on all processed powders, which were caused by plastic deformations induced by the impact of grinding media. It is also noticeable that starting from the processing time of 8 min and up to 16 min inclusive, some powder particles begin to acquire a discoid shape (Figure 4).

Figure 4. Images of nickel powder during its processing using 7 balls of 20 mm diameter for 8 min (a, b) and 12 min (c).

The investigation of nickel powders after MA with 6 balls of 20 mm diameter showed that the change in bulk density remains within the measurement error, except for the powders processed for 12 min (Figure 2). The change in bulk density of the powders
after 12 min of processing was 5.2%. However, changes in the apparent particle size are noticeable only when transitioning from 4 min of processing to 8 min (Figure 3). In this case, there is a jump of approximately 5%. Furthermore, the apparent particle size parameter remains almost unchanged thereafter. The formation of discoid nickel particles becomes noticeable starting from processing for 8 min (Figure 5). Additionally, at 16 min of processing, the quantity of discoid nickel particles is slightly higher than at 8 and 12 min of processing.

Figure 5. Images of nickel powder during its processing using 6 balls of 20 mm diameter for 8 min (a), 12 min (b), and 16 min (c).

The 5.2% change in bulk density observed after 12 min of processing may be linked to physical and chemical transformations within the powder, such as alterations in porosity or material compaction. Extended processing times could result in particle aggregation or shape changes, thereby affecting bulk density. Prolonged activation might cause particles to bind together, forming less homogeneous and denser structures. Regarding apparent particle size, the jump observed between 4 and 8 min of processing suggests significant changes in particle dimensions or aggregation at this point. During the initial 4 min, particle activation and interaction may cause an initial increase in size. After 8 min, more pronounced changes in particle shape might account for the observed data jump. The formation of discoid particles, which begins after 8 min of processing, indicates a notable shift in particle shape. This transformation could be due to intense mechanical forces that cause particles to evolve from spherical to discoid forms. As processing continues to 16 min, the ongoing reshaping and reorientation of particles may contribute to further development of discoid structures.

When MA nickel powders with a combination of grinding media consisting of 6 balls of 20 mm diameter and 8 balls of 10 mm diameter, the nature of the change in bulk density is somewhat different (Figure 2). A practically linear dependency of bulk density reduction with increasing MA [23] time is observed. The maximum change in bulk density was observed at MA for 16 min, amounting to 7.6%. This effect is preliminarily associated with an increase in the amount of discoid nickel after MA, which, in turn, can be explained by the increased contact area of the grinding media due to the addition of 8 balls of 10 mm diameter. Analysis of the apparent particle size (Figure 3) showed a decrease in this parameter starting from the 8-min mark. Subsequently, minor changes were observed, except at the 16-min mark, where a slight increase was noticeable. This is presumably due to the higher deformation of nickel and increased formation, compared to previous variants, of discoid nickel particles, as confirmed by SEM (Figure 6). Such particles have more difficulty passing through sieves during fractional analysis, compared to particles of round shape, contributing to the increase in the apparent particle size at the 16-min mark.
Thus, during the MA of powders, the greatest effect is observed at the maximum processing time (16 min), as confirmed by the analysis of the apparent particle size and the external appearance of the powder using electron microscopy. Visually, the effect involves the deformation of the powder and an increase in the proportion of discoid particles, leading to the highest energy accumulation. Nickel powder processed with a combination of grinding media [24,25] consisting of 6 balls of 20 mm diameter and 8 balls of 10 mm diameter undergoes significant deformation. No significant change in the chemical composition on the surface of nickel powders, depending on the MA time, was observed. As XRMA showed, the surface of nickel particles is partially covered with oxides, which is associated with the oxidation process during MA. The chemical composition of the surface of the powder particles averaged 96.8–98.4% Ni and 0.8–1.7% O2.

![Figure 6](image-url)  
**Figure 6.** Images of nickel powder during its processing using 6 balls of 20 mm diameter combined with 8 balls of 10 mm diameter for 16 min. (a) magnification ×500; (b,c) magnification ×1000.

### 3.2. Second Stage

At the second stage, deposition with nickel powder introduction into the weld pool, with and without MA, was conducted. The microstructure of the deposited metal with the introduction of nickel powder not subjected to MA consists of a solid solution alloyed with chromium, silicon, and carbon (Figure 7a). Primary needle-like chromium carbides are also present in the structure, located in the eutectic, along with niobium carbides formed due to the alloying of the deposited metal with components of the powder wire blend. When introducing nickel powder after MA into the deposited metal, the formation of martensite grains in the solid solution was detected, as well as changes in the amount of eutectic (Figure 7b–d).

Figure 7a shows the microstructure without mechanical activation, dendritic or cellular structures are visible, indicating that the metal solidified from a liquid phase, typical of casting or welding processes. The distribution of nickel appears relatively uniform throughout the microstructure but lacks the fine detail that might be expected with mechanical activation. The grain boundaries are more prominent and less refined, suggesting lower mechanical properties due to the absence of grain refinement. There may also be some porosity or inclusions visible, indicating a less homogeneous mixture and potential sites for mechanical weakness.

Figure 7b shows the microstructure with mechanical activation (MA) using 7 balls of 20 mm diameter, the grains appear more refined and uniform, suggesting that mechanical activation has broken down the original dendritic structure into finer grains. The nickel particles are likely more finely dispersed and possibly more homogenized within the matrix due to the mechanical activation. The grain boundaries seem less pronounced and more integrated, indicating improved mechanical properties. There appears to be less
porosity and fewer inclusions, suggesting a more homogeneous and mechanically robust microstructure.

![Image](a)

![Image](b)

![Image](c)

![Image](d)

Figure 7. Microstructure of deposited metal with introduction of nickel powder without MA (a) and with MA using 7 balls of 20 mm diameter (b), 6 balls of 20 mm diameter (c), and 6 balls of 20 mm diameter combined with 8 balls of 10 mm diameter (d).

Figure 7c shows the microstructure without mechanical activation using balls of 20 mm diameter, mixed grain structures are visible. The grains appear somewhat refined but not as uniformly as in the mechanically activated sample. This indicates that the use of balls of 20 mm diameter had some effect, but without mechanical activation, the refinement is incomplete. The distribution of nickel is less uniform compared to the mechanically activated sample, with some larger particles and clusters. The grain boundaries are more distinct compared to the mechanically activated sample, suggesting less integration and cohesion within the structure. Noticeable porosity and inclusions are present, indicating a less homogeneous mixture and potential sites for mechanical weakness.

Comparing both microstructures, it is evident that mechanical activation using 7 balls of 20 mm diameter has significantly improved the microstructure by refining the grain size, enhancing the distribution of nickel, and reducing porosity and inclusions. The microstructure in the image with MA is likely to have superior mechanical properties such as increased strength, hardness, and possibly better wear resistance compared to the non-activated sample.

Additionally, an increase in the hardness of the deposited metal was observed when introducing nickel powder after MA (Figure 8). Moreover, the higher the deformation of nickel powder after MA, the higher the hardness of the deposited metal. The variations in hardness of the deposited metal are directly linked to the MA process and the resulting deformation of nickel powder. The introduction of MA leads to a marked increase in hardness, with higher deformation corresponding to greater hardness. The configuration using 7 balls of 20 mm diameter produces the highest hardness, followed by 6 balls of 20 mm diameter and 8 balls of 10 mm diameter. This indicates that both the number and
size of the balls used in the MA process significantly affect the degree of deformation and the resulting hardness of the deposited metal. Further studies could investigate optimizing the combination of ball size and quantity to achieve the desired hardness level.

Figure 8. Hardness of deposited metal when introducing nickel powders with and without mechanical activation.

3.3. GA-Based CNN Hyperparameter Optimization for Surface Analysis

Mechanical activation alters the metal’s microstructure, impacting its physical and mechanical properties. Traditional methods for analyzing these changes are time-consuming and prone to subjective errors. DNNs can automatically process and analyze large volumes of microstructural images, identifying subtle features and correlations that may not be apparent to the human eye. This automated analysis helps establish a clear relationship between MA parameters and microstructural changes, providing insights into the underlying mechanisms and aiding in the optimization of processing techniques. Consequently, DNNs facilitate the development of predictive models that can forecast material properties based on their microstructure, streamlining the creation of new materials and enhancing their performance.

For instance, in the case of depositing nickel powder onto a metal surface without prior MA treatment, the microstructure typically consists of a solid solution alloy, doped with chromium, silicon, and carbon. However, introducing nickel powder after MA treatment induces changes in the microstructure, leading to the formation of martensite within the solid solution grains, along with alterations in the eutectic structure. Moreover, the hardness of the deposited metal increases after introducing nickel powder post-MA [26].

In this context, employing deep neural network (DNN)-based image processing techniques enable efficient and accurate analysis of microstructural images, allowing for the identification and quantification of various microstructural features, such as grain boundaries [27], carbide formations [28], and phase distributions [29]. By leveraging DNNs, it is possible to classify images of the microstructure with higher effects of MA treatment.

It was employed a genetic algorithm (GA) [30] to fine-tune the hyperparameters of a convolutional neural network (CNN) [31] used for surface analysis after MA. To prepare for this task, a dataset consisting of images depicting microstructure surfaces was compiled, as illustrated in Figure 7. These images were sliced into $100 \times 100$ pixel fragments to augment the dataset [32,33], resulting in a total of 1280 image fragments. Corresponding labels were also recorded to ensure accurate classification.
The dataset was divided into training and testing sets using stratified sampling, with 80% allocated for training and 20% for testing. This resulted in a train set of 1024 images and a test set of 256 images. It was utilized the DEAP [34] library to implement the GA, optimizing the following hyperparameters:

- Filter Sizes: The convolutional layers use filters of varying sizes denoted as $F_i$, where $i \in \{1, 2, \ldots, n\}$. For each filter size, the mathematical representation is given by:

$$\text{Filter Size} = F_i \text{ for } i \in \{1, 2, \ldots, n\}$$  \hspace{1cm} (3)

- Number of Units in Dense Layers: The dense layers consist of $U_j$ units, where $j$ varies depending on the layer. This is represented as:

$$\text{Number of Units} = U_j \text{ for } j \in \{1, 2, \ldots, m\}$$  \hspace{1cm} (4)

- Optimizer: The choice of optimizer, $O$, is one of several available options such as Adam, SGD, or RMSprop. The optimizer affects the gradient descent updates as follows:

$$\text{Update Rule} = \text{Optimizer}(\nabla L(\theta))$$  \hspace{1cm} (5)

where $\nabla L(\theta)$ denotes the gradient of the loss function $L$ with respect to the parameters $\theta$.

- Epochs: The number of epochs, $E$, used for training is varied. The training process iterates $E$ times as follows:

$$\text{Training Iterations} = E$$  \hspace{1cm} (6)

- Batch Size: The batch size, $B$, determines how many samples are processed before updating the model weights:

$$\text{Batch Size} = B$$  \hspace{1cm} (7)

The GA operated on a population of 10 individuals [35] across 5 generations [36]. The fitness function $F$ was based on the validation accuracy $A_v$ of the CNN model trained with the hyperparameters selected by the GA:

$$F = A_v$$  \hspace{1cm} (8)

where $A_v$ is the accuracy of the CNN on the validation set.

The CNN model architecture generated by the GA was defined using TensorFlow’s Keras API [37]. Variations included the number of convolutional layers $L_c$, max-pooling layers $L_p$, flattening layers $L_f$, and densely connected layers $L_d$. The architecture can be represented as:

$$\text{CNN Architecture} = \{L_c, L_p, L_f, L_d\}$$  \hspace{1cm} (9)

Activation functions used included ELU [38] or ReLU [39] for convolutional layers and ReLU for dense layers, applied as:

$$\text{Activation Function} = \text{ReLU or ELU}$$  \hspace{1cm} (10)

The output layer utilized a sigmoid activation function [40] for binary classification [41]:

$$\sigma(z) = \frac{1}{1 + e^{-z}}$$  \hspace{1cm} (11)

where $z$ represents the input to the output layer.

The best architecture and hyperparameters identified by the GA comprised 3 convolutional layers and 2 dense layers and depicted in Figure 9, with a corresponding validation accuracy of 0.94 achieved for distinguishing between microstructure images with and without MA treatment.
The inclusion of three convolutional layers aimed to extract relevant features from the microstructure images. The first convolutional layer captured basic features such as edges and textures, effectively preprocessing the input images. The second convolutional layer built upon these features, identifying more intricate patterns and shapes within the microstructures. The third convolutional layer further refined the feature maps, capturing high-level abstractions essential for accurate classification. This hierarchical feature extraction process significantly improved the network’s ability to differentiate between the nuanced differences in the microstructures caused by MA treatment.

Following the convolutional layers, the architecture included two dense layers. First of them layer consolidated the features extracted by the convolutional layers, enabling the network to learn complex combinations of these features and enhancing its discriminatory power. The second dense layer, serving as the output layer, provided the final classification decision. The use of a softmax activation function in this layer facilitated the conversion of learned features into probabilities, ensuring accurate predictions.

GA-based optimization approach effectively identified an optimal set of hyperparameters, enhancing the performance of the CNN. Key hyperparameters tuned by the GA included the learning rate, which balanced the speed and stability of the training process, and the batch size, which maintained a balance between training speed and model performance. GA also optimized filter sizes and counts for each convolutional layer, ensuring the network captured the most relevant features at each level of abstraction. Additionally, the choice of activation functions was tuned to ensure non-linearity and effective gradient flow during backpropagation, contributing to the network’s ability to learn complex patterns.

The validation accuracy of 0.94 indicates a high level of performance and generalization capability of the optimized CNN, suggesting that the network can reliably distinguish between microstructure images with and without MA treatment. This success highlights the potential of genetic algorithms (GA) in optimizing neural network architectures, particularly in specialized domains like microstructure analysis. The constructed confusion matrix
and the derived $F_1$ score of approximately 0.95 further validate the model’s robustness and its suitability for practical applications in microstructure image classification.

Figure 10 illustrates confusion matrix for the test set and shows the distribution of true positives, true negatives, false positives, and false negatives for the predicted classifications of microstructure images with and without MA treatment. The high values along the diagonal (156 and 85) indicate the model’s strong performance in correctly identifying the images, while the low values off the diagonal (10 and 5) reflect the relatively few misclassifications.

![Confusion Matrix for Test Set](image.png)

Figure 10. Confusion matrix for test set.

4. Discussion

CNNs are widely used for analyzing metal surface images in industrial quality control. They automatically extract and analyze image features, enabling the detection of defects such as cracks [42], corrosion [43], and dents [44]. The key advantages of CNNs include automatic feature extraction without manual intervention, resilience to variations in scale, lighting, and noise, and high accuracy in defect segmentation and classification. These attributes make CNNs suitable for real-world industrial environments. By developing models that automatically analyze and classify defects, CNNs enhance inspection speed, accuracy, cost efficiency, and reduce human error. This study shows a case of optimized CNN performance for these tasks with application of GA to fine-tune CNN hyperparameters. The three convolutional layers of the optimized network effectively extract hierarchical features from microstructure images, while the two dense layers enhance the classification capability. The resulting validation accuracy of 0.94 underscores the model’s robustness and its ability to generalize well to new data.

The metal additive manufacturing (AM) process can produce complex products with minimal waste but often requires post-processing due to poor surface quality. Abhilash et al. [45] combine CNN classification with electrical discharge-assisted post-processing to enhance surface quality. The CNN model, validated with five-fold cross-validation and achieving 96% accuracy, determines the polishing depth and passes. Low-energy polishing significantly improves the surface finish by 74% and reduces short-circuit discharges and elemental migration, resulting in a surface finish improvement from 97.3 to 12.62 µm.

Effective surface defect detection in metal structures requires the timely identification of defects. Konovalenko et al. [46] compare U-Net-like architectures with various encoders (ResNet, DenseNet, etc.) for defect detection. The best results are achieved using a U-Net with a ResNet152 backbone, optimized with stochastic gradient descent and Nesterov momentum, yielding a Dice Similarity Coefficient of 0.9304 and an IoU of 0.9122 on the test dataset.

The automatic inspection of metallic surface defects is crucial for industrial quality control but challenging due to complex conditions. Traditional methods often fail under varying scales, lighting, or noise. Tao et al.’s [47] paper introduces a novel cascaded autoencoder (CASAE) architecture for robust defect detection. The CASAE segments defects into pixel-wise masks and classifies them using a compact CNN. Tested on an
industrial dataset, this method proves accurate and robust for detecting metallic defects and is adaptable to other applications.

Further research directions may include optimization of the vibratory mill [48,49] processing parameters, such as rotation speed [50] and processing duration [50], to optimize the nickel powder mechanical alloying process. Additionally, the processing duration and mill loading parameters may affect the mechanical characteristics of nickel powder. This can be studied through hardness testing [51], strength testing [52], and other mechanical property assessments of processed samples. After gaining a deeper understanding of the vibratory mill’s impact on nickel powder structure and properties, its potential use in various industrial processes such as additive manufacturing, powder metallurgy, and others can be explored.

However, it is important to consider the following limitations such as the physical limitations of the vibratory mill because some parameters, such as particle size and shape, may be constrained by the physical characteristics of the equipment. [53] Research aimed at optimizing the process should also consider equipment costs and energy consumption for nickel powder processing.

When working with the optimization of hyperparameters in CNN using GA [54], several limitations and challenges may arise, including significant computation time, especially with large datasets and complex CNN models. Additionally, running GA with a large number of individual models and generations can require substantial computational resources [55], including CPU time and memory. Since GA uses a stochastic approach to optimization, results can be uncertain and dependent on random factors. Sometimes it is not always easy to choose suitable hyperparameters for optimization, and improper selection can lead to underfitting or overfitting [56] of the CNN model and hyperparameter space of CNNs can be very large, making it difficult to fully explore all possible options and potentially leading to suboptimal solutions. Despite these limitations, using GA for hyperparameter optimization in CNNs also offers significant prospects.

As a benefit, GA allows for the automation of the hyperparameter selection process [57], freeing the researcher from the need for manual tuning and can efficiently and quickly find optimal hyperparameters for the CNN model. Since GA is based on an evolutionary approach, the optimal hyperparameters found may have the ability to generalize to other datasets and tasks [58]. Also, properly tuned hyperparameters can improve the performance of the CNN model, including accuracy and training speed. Given these limitations and prospects, the use of GA for optimizing hyperparameters in CNNs is a powerful tool in the field of machine learning and deep learning research.

5. Conclusions

Based on the conducted study, several key insights have emerged regarding the mechanical activation (MA) of nickel powder and its subsequent applications. The research has provided valuable information on the effectiveness of vibratory milling for MA, the impact of different processing parameters, and the potential improvements in surface analysis methodologies. The following points summarize the main findings:

1. Viability of Vibratory Mill for Mechanical Activation (MA): The study confirms that using a vibratory mill for MA of nickel powder is effective. Extending the processing duration is recommended to optimize results.
2. Particle Deformation: Nickel powder particles exhibit significant deformation, with discoid particles observed at a processing time of 16 min. The best outcomes are achieved using a mill with a grinding media mix of 6 balls (20 mm diameter) and 8 balls (10 mm diameter). Processing also results in reduced bulk density of the powder.
3. Impact on Weld Pool: Incorporating MA nickel powder into the weld pool shows minimal effects on the metal structure but leads to increased hardness. Further investigation is needed to explore the structural and property changes under conditions of greater damage and deformation.
4. Optimization of Convolutional Neural Networks (CNNs): The study highlights the effectiveness of genetic algorithms (GAs) in optimizing CNNs for analyzing surfaces with and without MA, suggesting promising advancements in surface analysis techniques.

These findings provide a foundation for further research into optimizing MA processes and enhancing the capabilities of surface analysis methodologies.

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