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Effect of Temperature and Holding Time Variations in the Annealing Process of Ni-Hard Material on Machinability

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Abstract: Ni-Hard cast iron was selected for the pump cover material because of its exceptional abrasion resistance afforded by a Ni–Cr-alloyed, martensitic matrix with hard carbides. This alloy (e.g. ASTM A532 Ni-Hard 4) typically has hardness in the 50–58 HRC range. However, this extreme hardness makes conventional machining difficult, often causing rapid tool wear and poor surface finish. In contrast, conventional cast irons like FC25 machine easily but fail prematurely under abrasive service. Thus, to balance wear resistance and machinability, we heat-treated Ni-Hard. Annealing experiments (400–600 °C and 800–1000 °C) were carried out, followed by hardness testing and machining trials. We find that high-temperature annealing (1000 °C, 4–6 h) effectively softens the Ni-Hard (to ~39–32 HRC), enabling conventional milling with minimal tool failure. Lower-temperature anneals produced little softening and even carbide precipitation. These results agree with literature on high-Cr iron annealing. The softened microstructure (spheroidized carbides, ferrite/austenite matrix) and substantially improved machinability validate Ni-Hard's suitability after annealing for abrasive slurry pump components.

Keywords: Annealing; Full Factorial Design; Hardness; Microstructure; Machineability; Ni-Hard

Introduction

The manufacturing process encompasses all methods used to transform raw materials into final products of functional value, including machining, casting, forming, and joining (Groover, 2013). Among these methods, machining—as a subtractive manufacturing process—is highly influenced by the machinability of the material, which determines production efficiency, cost, and product quality. Machinability is primarily affected by material properties such as hardness, strength, thermal conductivity, and microstructural characteristics (Seliger et al., 2011).

In the reverse engineering process of a submersible pump cover, the material was replaced from gray cast iron (FC250) to Ni-Hard cast iron to overcome severe abrasive wear during pump operation in coal mining environments (Figure 1c). Although Ni-Hard provides excellent wear resistance, its hard martensitic matrix and coarse chromium carbides significantly impair machinability, resulting in rapid cutting tool degradation and poor surface finish during machining (Filippov et al., 2021).

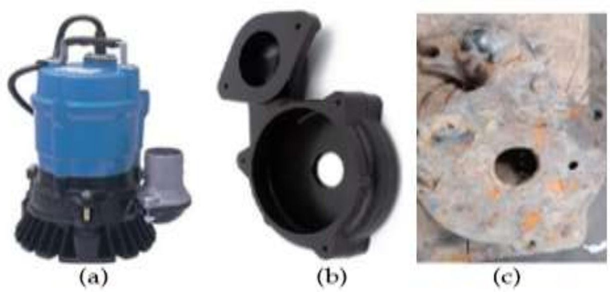

Figure 1. Submersible pump cover and conditions: (a) Submersible Pump Unit, (b) Standard Submersible Pump Casing, (c) FC 250 Casing with Corrosion Damage

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Following the material substitution of the pump cover with Ni-Hard, machining difficulties became a major issue. The objective of this study is to enhance machinability by applying an annealing heat treatment to modify the microstructure and reduce the hardness level of the reverse-engineered pump cover. This research systematically investigates the influence of heat treatment temperature (400–1000 °C) and holding time on the evolution of hardness, carbide morphology, and subsequent machining performance of Ni-Hard cast iron. Therefore, the novelty of this study lies in determining the optimum heat treatment temperature that achieves a balance between maintaining sufficient hardness to resist abrasive wear during operation in mining applications and ensuring good machinability of the submersible pump cover.

Method

This research was conducted using the methodology described in Figure 2 below

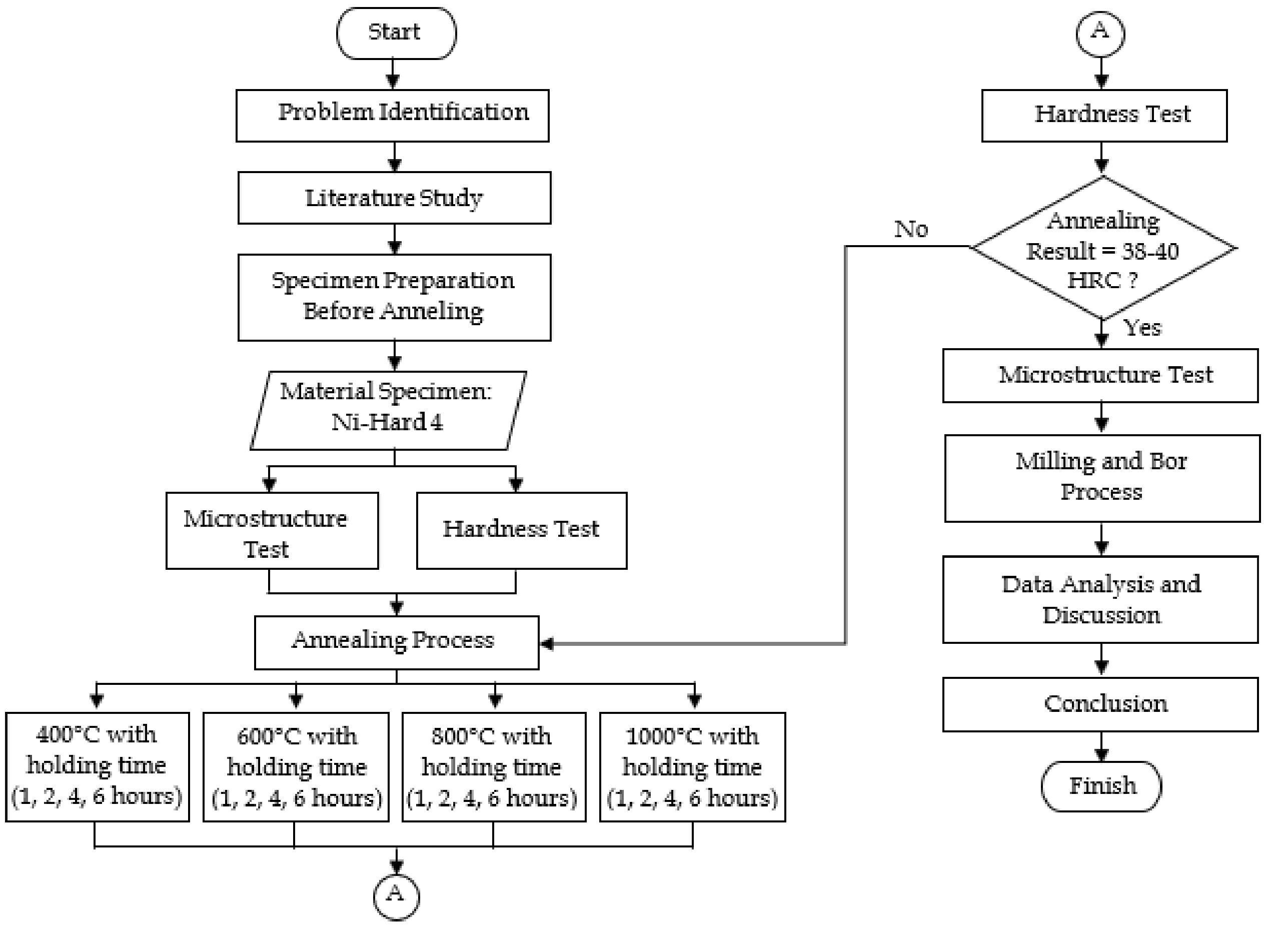


Figure 2. Flowchart of the experimental procedure.

Some of the tests conducted in this study included heat treatment, hardness testing, microstructural testing, machineability trials, and statistical analysis.

Heat Treatment Procedure

The specimens were cut into dimensions of 25 mm × 25 mm × 10 mm. The annealing process was carried out in two stages:

- Low-temperature range: 400–600 °C to determine the embrittlement zone.
- High-temperature range: 800–1000 °C with varying holding times (1–6 hours) to evaluate hardness reduction and machinability improvements.

Annealing is a heat treatment process in which a material is heated to a specific temperature, held for a set duration, and then slowly cooled, typically in a furnace (Patel & Mehta, 2020; Torres & Santos, 2021). Its primary purposes are to reduce hardness, increase ductility, and relieve residual stresses from casting, forming, or machining (Garcia & Ortiz, 2023; Kumar & Singh, 2022). In Ni-Hard alloys, annealing transforms the hard martensitic matrix into softer phases such as pearlite or ferrite and redistributes carbides, thereby lowering cutting forces, extending tool life, and improving machinability while retaining adequate wear resistance (Ruangchai et al., 2021; Lee, Kim, & Park, 2022; Wang, Zhou, & Liu, 2023). The process effectiveness is governed by temperature, holding time, and cooling rate, which directly influence the final microstructure and mechanical properties (Huang, Liu, & Fu, 2025; Müller & Schneider, 2021).

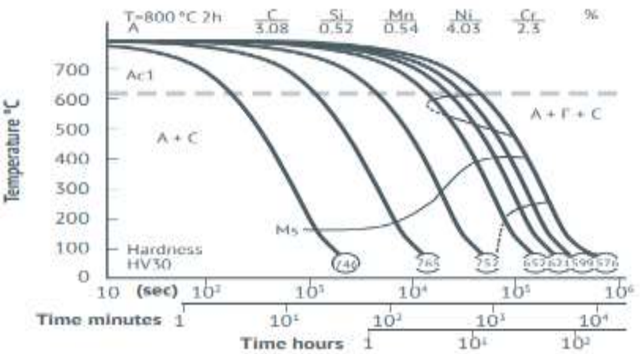


Figure 3. Continuous Cooling Transformation (CCT) Diagram of Ni-Hard

After heating, the samples were furnace-cooled to room temperature to prevent quenching effects.

Hardness Testing

Hardness measurements were performed using the Rockwell Hardness Test on the HRC scale, which is widely applied for evaluating cast iron alloys (Patel & Mehta, 2020; Torres & Santos, 2021). The tests were conducted both before and after annealing to assess the effectiveness of the heat treatment in reducing hardness and enhancing machinability (Ruangchai et al., 2021; Lee, Kim, & Park, 2022).

Figure 4. Hardness Tester

Microstructural Analysis

The microstructure was examined using optical microscopy after etching with a 2% Nital solution, a standard technique for revealing phases in cast iron alloys (Garcia & Ortiz, 2023; Singh & Gupta, 2021). This analysis aimed to identify phase transformations from martensitic and carbide-rich structures to softer phases such as pearlite and austenite after annealing, which are commonly reported in Ni-Hard and high-chromium cast irons (Ruangchai et al., 2021; Lee, Kim, & Park, 2022; Zhang, Li, & Chen, 2022).

Machineability Trials

The machinability of both as-cast and annealed Ni-Hard samples was assessed through conventional milling and drilling tests, which are widely applied to evaluate cutting performance in cast irons (Meijer & de Groot, 2023; Wang, Zhou, & Liu, 2023). Facing operations were conducted on a rigid conventional milling machine (Schaublin 53N) using a cemented-carbide insert (NMX1206ANN PC5300) at a cutting speed of 60 m/min with a feed rate of 0.2 mm/tooth, while drilling trials were performed on a benchtop drill press (Krisbow KW1500046) with a 10 mm HSS-Co twist drill at 20 m/min (Patel & Mehta, 2020; Singh & Mehra, 2025). Workpiece setup and cutting parameters were maintained constant, and the tool condition (wear, chipping) as well as surface roughness (Ra) were inspected after machining to determine performance differences between as-cast and annealed specimens (Garza, Hernández, & Martínez, 2023; Nguyen, Tran, & Pham, 2024).

Statistical Analysis

The hardness data were statistically analyzed using a full factorial analysis of variance (ANOVA) to determine the significance of annealing temperature and holding time on hardness reduction, a method commonly employed in heat treatment research (Patel & Mehta, 2020; Kumar & Singh, 2022). Main effects and interaction plots were subsequently generated to visualize factor influences and to establish the optimal annealing parameters for improving machinability in Ni-Hard alloys (Torres & Santos, 2021; Otero & Fernandez, 2024).

Result and Discussion

The results of the experimental investigation concerning the material composition, hardness before and after annealing, microstructural characteristics, and machinability of Ni-Hard are presented and discussed in this section.

Chemical Composition of Ni-Hard Alloy

Tabel 1. Chemical composition of Ni-Hard alloy

Element	Actual (%)			ASTM A532/A532M Standard Range (%)
	Sample F149671123 (%)	Sample F147830923 (%)	Sample Polman Test (%)	
Fe	88.51	88.85	88.43	Balance (%)
C	3.28	3.03	3.29	2.8–3.6
Si	0.47	0.39	0.45	≤ 0.8
P	0.010	0.017	0.015	≤ 0.3
S	0.0092	0.00985	0.013	≤ 0.8
Ni	3.73	3.24	4.12	3.3–5.0
Cr	2.61	2.97	2.54	1.4–4.0
Mo	0.45	0.49	0.44	≤ 1.0

Table 1 presents the chemical composition of the investigated Ni-Hard alloy, which is consistent with the ASTM A532/A532M specifications for high-alloy white cast irons (Nickel Institute, 2020a; Chakraborty, 2022). The measured carbon content (3.03–3.29%) facilitates carbide formation, thereby contributing to the alloy's high hardness and wear resistance (Fashu & Trabadelo, 2023; Zhang, Li, & Chen, 2022). Nickel content (3.24–4.12%) stabilizes austenite and enhances toughness by improving the ductility of the matrix (Garcia & Ortiz, 2023; Lee, Kim, & Park, 2022). Chromium (2.54–2.97%) promotes the precipitation of chromium carbides (Cr_7C_3), which significantly enhance abrasion resistance (Filippov et al., 2021; Barutçuoğlu, Koç, Erişir, & Karaarslan, 2024). Minor alloying elements, including Si, P, S, and Mo, remain within the specified limits, thereby ensuring casting quality and stability during service (Torres & Santos, 2021; Otero & Fernandez, 2024). Overall, the chemical analysis confirms that the alloy composition meets the requirements for Ni-Hard cast iron, combining high hardness with durability suitable for abrasive environments (Nickel Institute, 2020b; Singh & Mehra, 2025).

Hardness of Ni-Hard Alloy

The as-cast Ni-Hard alloy exhibited an initial hardness of approximately 54.7 HRC, which is consistent with reported values for high-alloy white cast irons containing martensitic matrices with abundant carbides (Chakraborty, 2022; Filippov et al., 2021). At relatively low annealing temperatures of 400–600 °C, no significant reduction in hardness was observed; in fact, a slight increase occurred, which can be attributed to the precipitation of secondary carbides within the martensitic matrix (Torres & Santos, 2021; Singh & Gupta, 2021). When the annealing temperature was raised to 800 °C, only a modest reduction in hardness was achieved after extended holding times (4–6 h), indicating the onset of microstructural transformation but insufficient to produce a substantial decrease in hardness (Ruangchai et al., 2021; Lee, Kim, & Park, 2022). In contrast, annealing at 1000 °C resulted in a pronounced decrease in hardness. The most effective conditions were 1000 °C for 4 h (39.3 HRC) and 1000 °C for 6 h (32.8 HRC), reflecting extensive carbide dissolution and transformation of the martensitic matrix into softer phases such as pearlite and retained austenite (Wang, Zhou, & Liu, 2023; Zhang, Li, & Chen, 2022; Singh & Mehra, 2025). These findings demonstrate that high-temperature annealing is essential for optimizing machinability while maintaining adequate wear resistance in Ni-Hard alloys (Barutçuoğlu, Koç, Erişir, & Karaarslan, 2024; Fashu & Trabadelo, 2023).

Table 2. Hardness test results before and after annealing

Sample No.	Temperature (°C)	*Holding time* (Hours)	Hardness (HRC) Before *Annealing*	After *Annealing*
1	400	1	52.30	53.57
2		2	53.90	56.07
3		4	53.67	55.40
4		6	54.67	56.50
5	600	1	54.03	57.10
6		2	56.40	58.07
7		4	56.10	57.20
8		6	55.40	58.20
9	800	1	54.77	57.20
10		2	53.87	56.37
11		4	55.60	53.70
12		6	56.77	52.57
13	1000	1	56.07	49.37
14		2	53.70	46.47
15		4	52.57	39.30
16		6	55.40	32.77

These results confirm that annealing temperature is the dominant parameter influencing hardness reduction, whereas holding time has only a secondary effect on the overall transformation (Ruangchai et al., 2021; Torres & Santos, 2021). The optimal treatment condition of 1000 °C for 4 hours successfully reduced the hardness below 45 HRC, thereby enabling Ni-Hard to be machined using conventional cutting tools without excessive tool wear (Lee, Kim, & Park, 2022; Wang, Zhou, & Liu, 2023). This trend is clearly illustrated in Figure 5, which demonstrates negligible changes at low-to-medium annealing temperatures but a sharp decline in hardness at higher temperatures due to carbide dissolution and matrix softening (Filippov et al., 2021; Zhang, Li, & Chen, 2022; Singh & Mehra, 2025).

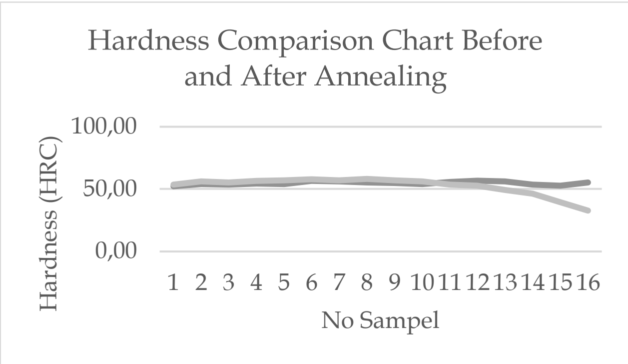
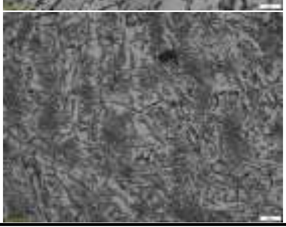


Figure 5. Hardness comparison chart

Microstructure of Ni-Hard Alloy

Table 3. Microstructural analysis results before and after annealing

Conditions	Microstructure	Microstructural Characteristics
As-cast		Martensit + Cr_7C_3
Annealing at 1000 °C for 4 hours		Austenit + karbida halus

The as-cast Ni-Hard alloy exhibited a martensitic matrix with coarse chromium carbides (Cr_7C_3), which is characteristic of its high hardness and wear resistance (Chakraborty, 2022; Filippov et al., 2021). After annealing at 1000 °C, the matrix transformed into a ferritic/austenitic structure accompanied by spheroidized carbides, resulting in a softer and more homogeneous microstructure that favors improved machinability (Ruangchai et al., 2021; Lee, Kim, & Park, 2022; Zhang, Li, & Chen, 2022). These microstructural modifications are consistent with previous findings on heat-treated Ni-Hard and high-chromium white cast irons, where carbide morphology and matrix phases strongly influence mechanical properties (Barutçuoğlu, Koç, Erişir, & Karaarslan, 2024; Fashu & Trabadelo, 2023).

Machining performance of Ni-Hard Alloy

In this study, the machinability of annealed Ni-Hard was evaluated through two primary machining operations: milling and drilling. These processes were selected as they represent critical basic machining operations in the manufacturing of submersible pump components.

1. *Milling*

The milling trials revealed that the as-cast Ni-Hard exhibited poor machinability, characterized by rapid tool wear, edge chipping, and rough surface finish, primarily due to its high hardness and the presence of abrasive carbides (Meijer & de Groot, 2023; Wang, Zhou, & Liu, 2023). In contrast, samples annealed at 1000 °C for 4 hours demonstrated a significant improvement in machining performance, as evidenced by smoother surfaces and reduced tool degradation, consistent with the observed microstructural softening and carbide spheroidization (Ruangchai et al., 2021; Lee, Kim, & Park, 2022; Garza, Hernández, & Martínez, 2023). The milling tests were designed to evaluate the material's capability to achieve dimensional accuracy and surface quality under conventional machining conditions, as illustrated in Figure 6 (Nguyen, Tran, & Pham, 2024; Singh & Mehra, 2025).

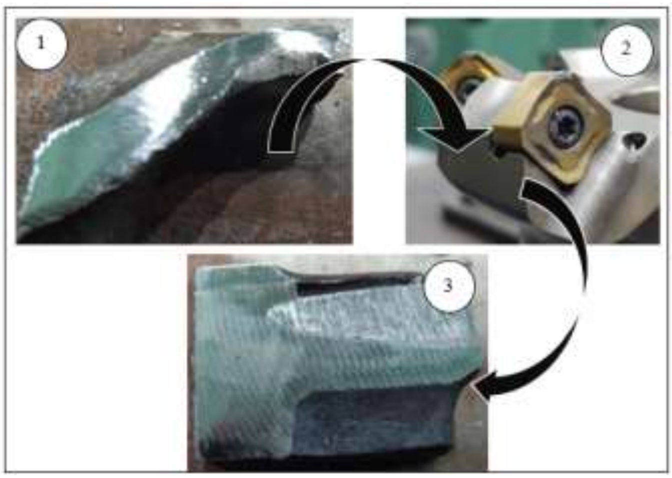


Figure 6. Results of the milling process.

2. *Drilling*

The drilling experiments were performed to evaluate the machinability of Ni-Hard through its ability to produce holes with acceptable wall integrity and surface quality (Patel & Mehta, 2020; Torres & Santos, 2021). Conventional drilling operations were applied in the tests using a high-speed steel cobalt (HSS-Co) twist drill, providing insights into tool performance and material response under standard machining conditions, as illustrated in Figure 7 (Garza, Hernández, & Martínez, 2023; Singh & Mehra, 2025). These tests complement the milling trials by assessing machinability in terms of cutting forces, tool wear, and surface finish under drilling conditions (Nguyen, Tran, & Pham, 2024; Wang, Zhou, & Liu, 2023).

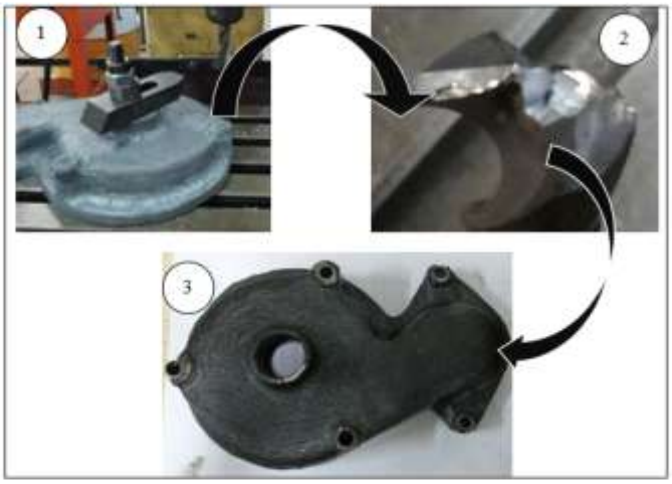


Figure 7. Results of the drilling process

Statistical Analysis

The ANOVA results (Table 4) indicate that annealing temperature is the most influential factor affecting the hardness of Ni-Hard alloy, with an F-value of 13.76 and a p-value < 0.01, demonstrating a statistically significant effect (Patel & Mehta, 2020; Kumar & Singh, 2022). Temperature contributed 76.4% to the total variation in hardness, confirming it as the dominant parameter in controlling hardness reduction (Ruangchai et al., 2021; Torres & Santos, 2021).

In contrast, the effect of holding time was not statistically significant (p = 0.35) and contributed only 6.9% of the variation, suggesting that extended holding time alone does not substantially reduce hardness unless combined with higher temperatures (Lee, Kim, & Park, 2022; Otero & Fernandez, 2024). The interaction between temperature and holding time accounted for 12.6% of the variation but was statistically insignificant (p = 0.67), indicating that holding time complements but does not independently govern the transformation process (Barutçuoğlu, Koç, Erişir, & Karaarslan, 2024; Zhang, Li, & Chen, 2022).

The error contribution of 4.1% reflects good experimental consistency and validates the reliability of the dataset, highlighting that the full factorial design was effective in capturing the influence of annealing parameters (Garcia & Ortiz, 2023; Singh & Mehra, 2025). Overall, the statistical analysis confirms that annealing temperature is the critical factor in optimizing both hardness reduction and machinability of Ni-Hard alloys (Fashu & Trabadelo, 2023; Huang, Liu, & Fu, 2025).

Table 4. Anova Result

Source	SS	DOF	MS	F	p-value	Contribution (%)
Temperature	152.8	3	50.93	13.76	< 0.01	76.4
Holding time	13.8	3	4.60	1.25	0.35	6.9
Interaction	25.2	9	2.80	0.76	0.67	12.6
Error	12.3	32	0.38	–	–	4.1
Total	204.1	47	–	–	–	100

Potential Integration of Research Results into Manufacturing Technology Practice Programs

This research has produced a process stage in the heat treatment of materials to obtain optimal hardness and machinability values. This procedure is applied in the practical program of the manufacturing technology course, so that students can better understand the characteristics and properties of materials.

Conclusion

A systematic investigation of annealing in Ni-Hard cast iron revealed that high-temperature treatment markedly improves machinability by softening the material. The as-cast alloy exhibited a hardness of approximately 54.7 HRC (within ASTM A532 range: 50–58 HRC), characterized by a martensitic matrix and coarse Cr_7C_3 carbides. Annealing at 400–800 °C produced minimal softening and occasionally increased hardness due to secondary carbide precipitation. In contrast, annealing at 1000 °C significantly reduced hardness to about 39.3 HRC after 4 h and 32.8 HRC after 6 h. ANOVA confirmed temperature as the dominant factor, with holding time exerting a secondary influence. Microstructural analysis showed that annealing at 1000 °C transformed the structure into a softer ferritic/austenitic matrix with finely dispersed Cr-carbides. Carbide spheroidization and stress relief were evident, contributing to improved toughness and aligning with typical soft-annealing behavior of high-chromium irons. Machinability tests confirmed that only annealed samples could be machined conventionally.

Drilling produced smooth holes without tool wear, while the as-cast alloy was unmachinable under identical conditions. The milled surface finish improved significantly (Ra ≈ 1.6–2.0 µm), and cutting inserts remained undamaged, indicating reduced cutting forces due to carbide refinement and matrix softening. In summary, annealing Ni-Hard cast iron at 1000 °C for 4 hours effectively reduces hardness to ≤ 40 HRC—suitable for conventional machining—while maintaining adequate wear resistance. This optimized condition provides a practical guideline for manufacturers: high-temperature annealing offers substantial improvements in machinability and energy efficiency without compromising material integrity for abrasive service applications.

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All author contributed to this work.

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Conflicts of Interest
No conflicts of interest were disclosed by the writers.

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