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Multi-Response Optimization of Cryogenic Treatment for EN Series Steels using Taguchi Method

Mr. Sagar Shivaji Sonawane[1], Dr. C. S. Padmavat[2], Dr. Abhay Pawar[3], Prof. S. B. Janjal[4], Prof. A. B. Humbe[5]
[1]*Master of Engineering Scholar, Department of Mechanical Engineering, International Center of Excellence in Engineering & Management, Chhatrapati SambhajiNagar, Maharashtra, India.*
[2]*Campus Director, International Center of Excellence in Engineering & Management, Chhatrapati SambhajiNagar, Maharashtra, India.*
[3,4,5]*Professor, Department of Mechanical Engineering, International Center of Excellence in Engineering & Management, Chhatrapati SambhajiNagar, Maharashtra, India.*

Abstract: Deep cryogenic treatment (DCT) has emerged as a supplementary process to conventional heat treatment for enhancing the mechanical and tribological properties of engineering steels. However, its systematic evaluation for commonly used EN series steels remains insufficiently explored. This study presents a comprehensive comparative investigation of the effect of DCT on the hardness and wear resistance of EN08, EN24, and EN31 steels using Taguchi L18 orthogonal array design. The specimens were subjected to conventional hardening and tempering, followed by deep cryogenic treatment at -196°C for 48 hours. Rockwell C hardness testing and Pin-on-Disk wear testing (ASTM G99) were conducted. The results revealed that DCT improved hardness in all three materials: EN08 from 50 to 54 HRC (8.0% improvement), EN24 from 53 to 55 HRC (3.8% improvement), and EN31 from 56 to 59 HRC (5.4% improvement). Wear testing showed mass loss ranging from 0.040 g to 0.100 g across 18 experiments. Taguchi S/N ratio analysis identified EN31 with DCT as the optimal combination for maximizing hardness, while ANOVA revealed Material (72.5%) and Treatment (20.1%) as the most significant factors influencing hardness. The study demonstrates that cryogenic treatment is an effective method for enhancing hardness of EN series steels, with material-dependent response, and provides quantifiable data for industrial adoption.
Keywords: Deep Cryogenic Treatment; EN Series Steels; Taguchi Method; Hardness; Wear Resistance; ASTM G99; Optimization; ANOVA.

I. INTRODUCTION

Wear is one of the most prevalent and costly forms of material degradation in industrial machinery, leading to reduced component efficiency, increased maintenance costs, and premature failure. The economic impact of wear is substantial, with wear-related losses accounting for a significant portion of industrial maintenance budgets worldwide. Improving the wear resistance of engineering components is therefore a critical objective for researchers and manufacturers alike [1].

Among engineering materials, EN series steels—EN08 (medium carbon), EN24 (nickel-chromium-molybdenum), and EN31 (high carbon chromium)—are widely used in automotive, bearing, and tooling applications due to their excellent combination of strength, toughness, and hardenability [2]. Conventional heat treatment (austenitizing, quenching, and tempering) is the standard industrial practice for enhancing their mechanical properties. However, conventional quenching inevitably leaves a fraction of retained austenite—a soft, metastable phase—within the martensitic matrix. This retained austenite can transform under service stresses, leading to dimensional instability, reduced wear resistance, and premature failure [3].

Cryogenic treatment has emerged as a promising supplementary process to address this limitation. Deep cryogenic treatment (DCT) involves cooling the material to temperatures below -150°C, typically using liquid nitrogen (-196°C), holding it at this temperature for an extended period (soaking), and gradually warming it back to room temperature [4]. This deep-freezing process facilitates the transformation of retained austenite to martensite and promotes the precipitation of fine, uniform secondary carbides (eta-carbides) within the martensitic lattice [5]. These microstructural refinements result in enhanced hardness, superior wear resistance, and improved dimensional stability [6, 7].

Research has shown that DCT can improve wear resistance by 50-300% in tool steels [4, 8]. Studies on EN31 bearing steel have demonstrated significant improvement in wear resistance after DCT [9, 10]. However, systematic comparative studies on commonly used EN series steels—EN08, EN24, and EN31—under identical processing and testing conditions are limited in the literature.

The Taguchi method, developed by Dr. Genichi Taguchi, provides a powerful framework for designing experiments and optimizing processes. It uses orthogonal arrays to efficiently study the effects of multiple factors with minimal experiments, and Signal-to-Noise (S/N) ratios to measure quality [11]. This method has been successfully applied in materials processing optimization [12, 13].

This study aims to systematically investigate and compare the effect of deep cryogenic treatment on the hardness and wear resistance of EN08, EN24, and EN31 steels using Taguchi L18 orthogonal array design, and to identify the optimal parameters for maximizing hardness and minimizing wear.

II. MATERIALS AND METHODS

A. Materials Selection

Three grades of EN series steels were procured from Jai Bhadra Metals, Waluj, Aurangabad, with Mill Test Certificates confirming their chemical composition as per BS 970 standards. The materials were supplied in the form of 16 mm diameter round bars.

Table 1: Chemical Composition of EN08, EN24, and EN31 Steels

Element	EN08 (%)	EN24 (%)	EN31 (%)	Specification
Carbon (C)	0.45	0.40	1.05	BS 970
Manganese (Mn)	0.65	0.63	0.52	BS 970
Chromium (Cr)	–	1.14	1.45	BS 970
Nickel (Ni)	–	1.45	–	BS 970
Molybdenum (Mo)	–	0.26	–	BS 970
Silicon (Si)	0.28	0.26	0.27	BS 970
Sulfur (S)	0.032	0.024	0.025	≤ 0.050
Phosphorus (P)	0.028	0.016	0.030	≤ 0.050

The disk used in the Pin-on-Disk wear testing setup was made of HARDOX 500 steel, a high-strength abrasion-resistant steel with a nominal hardness of 500 HBW (approximately 48-52 HRC), manufactured by SSAB.

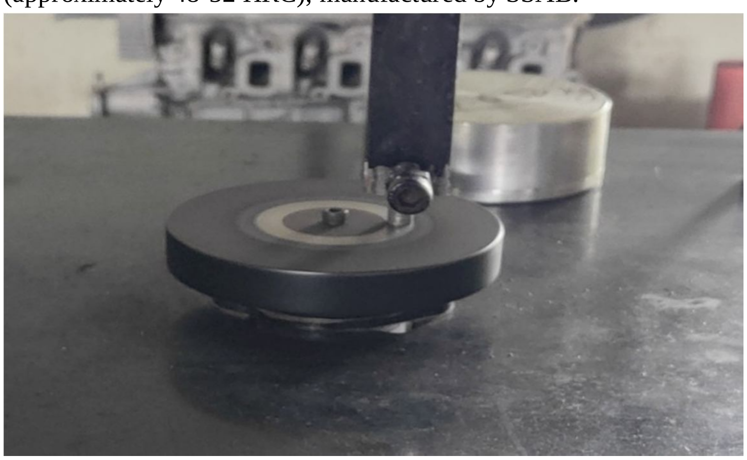

Figure 1: Disk (HARDOX 500)

B. Specimen Preparation

Pin specimens of 10 mm diameter × 32 mm length with flat tips were machined as per ASTM G99-17 specifications. The flat tip geometry was chosen to simplify machining and measurement, with wear volume calculated using the mass loss method. All specimens were machined at Mangaldeep Engineering College Workshop, Nipani, and surface finish of Ra ≤ 0.8 μm was achieved on the contact surfaces.

Figure 2: Specimen Machining

C. *Conventional Heat Treatment*

All 18 pin specimens were subjected to conventional heat treatment (hardening and tempering) at SN Heat Treatment Pvt. Ltd., Waluj. The heat treatment parameters for each steel grade are presented in Table 2.

Table 2: Conventional Heat Treatment Parameters

Steel Grade	Austenitizing Temperature (°C)	Austenitizing Time	Quenching Medium	Tempering Temperature (°C)	Tempering Time
EN08	830–860	45 min	Water/Oil	400–450	2 h
EN24	820–840	60 min	Oil	550–600	2 h
EN31	800–820	45 min	Oil	180–225	2 h

Figure 3: Conventional Heat Treatment Workshop

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D. Deep Cryogenic Treatment

After conventional heat treatment, the 18 pins were divided into two groups:

- Group A (CT): 9 pins (3 of each material) – Conventional treatment only (hardening + tempering)
- Group B (DCT): 9 pins (3 of each material) – Conventional treatment + deep cryogenic treatment + post-cryo tempering

The deep cryogenic treatment was carried out at AJ Smart Technologies, Waluj, Aurangabad. The process parameters are presented in Table 3.

Table 3: Deep Cryogenic Treatment Parameters

Parameter	Specification
Treatment Type	Deep Cryogenic Treatment (DCT)
Cooling Medium	Liquid Nitrogen (LN_2)
Target Temperature	−196°C
Cooling Rate	1–2°C/min
Soaking Duration	48 hours
Warming Rate	1–2°C/min
Final Temperature	Room Temperature
Post-Cryogenic Tempering	As per material specification (3 hours)

Figure 4: Cryogenic Treatment Workshop

E. Taguchi Design of Experiments

The Taguchi L18 orthogonal array was selected to design the experiments. Three factors were considered:

Table 4: Factors and Levels for Taguchi L18 Array

Factor	Level 1	Level 2	Level 3
A: Material	EN08	EN24	EN31
B: Treatment	CT	DCT	–
C: Load (Added Weight)	2 kg	3 kg	4 k

Table 5: Taguchi L18 Experimental Layout

Exp. No.	Material	Treatment	Added Weight	Total Pan Weight	Effective Force (N)
1	EN08	CT	2 kg	3.700 kg	76.8
2	EN08	CT	3 kg	4.700 kg	97.6
3	EN08	CT	4 kg	5.700 kg	118.3
4	EN24	CT	2 kg	3.700 kg	76.8
5	EN24	CT	3 kg	4.700 kg	97.6
6	EN24	CT	4 kg	5.700 kg	118.3
7	EN31	CT	2 kg	3.700 kg	76.8
8	EN31	CT	3 kg	4.700 kg	97.6
9	EN31	CT	4 kg	5.700 kg	118.3
10	EN08	DCT	2 kg	3.700 kg	76.8
11	EN08	DCT	3 kg	4.700 kg	97.6
12	EN08	DCT	4 kg	5.700 kg	118.3
13	EN24	DCT	2 kg	3.700 kg	76.8
14	EN24	DCT	3 kg	4.700 kg	97.6
15	EN24	DCT	4 kg	5.700 kg	118.3
16	EN31	DCT	2 kg	3.700 kg	76.8
17	EN31	DCT	3 kg	4.700 kg	97.6
18	EN31	DCT	4 kg	5.700 kg	118.3

The effective load on the pin was calculated using the principle of moments: Force on **Pin = (Pan Weight + Added Weight) × 9.81 × (L2/L1)**, where L1 = 26 cm (pin to pivot) and L2 = 55 cm (pan to pivot).

F. Hardness Testing

Hardness measurements were carried out using a Rockwell C hardness tester (Model: L-3 Rockwell cum Brinell Hardness Tester) at SN Heat Treatment Pvt. Ltd., Waluj. Five indentations were taken on each specimen at well-spaced locations (minimum 3 mm apart), and the average value was recorded. The test parameters were: diamond cone indenter (Brale), minor load of 10 kgf, major load of 150 kgf, and dwell time of 15 seconds.

Figure 5: Rockwell C Hardness Testing Machine

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G. Wear Testing (Pin-on-Disk)

Dry sliding wear tests were conducted on a manually operated Pin-on-Disk tribometer available in the ICEEM Workshop as per ASTM G99-17 standard. The test parameters were: constant hydraulic speed setting (same for all experiments), run time of 10 minutes per pin, track radius of 25-30 mm, HARDOX 500 disk (130 mm diameter, 20 mm thickness), laboratory air environment (room temperature), and dry sliding condition.

Figure 6: Pin-on-Disk Setup

Wear Measurement:

Wear was measured using the mass loss method. The pins were weighed before and after each test using a precision balance (0.001 g accuracy). The wear volume was calculated using:

$$\mathbf{V = (W\_initial - W\_final) / \rho}$$

Where:

- V = Wear volume (mm^3)
- W_initial = Initial weight (g)
- W_final = Final weight (g)
- ρ = Density of steel = 7.85 g/cm^3

III. RESULTS AND DISCUSSION

A. Hardness Results

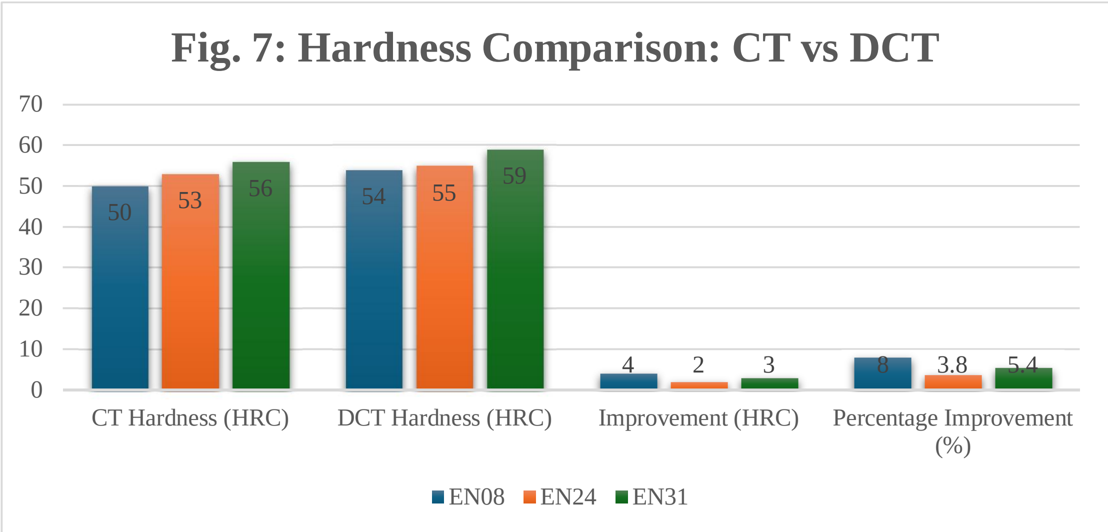
The hardness test results for CT and DCT specimens are presented in Table 6.

Table 6: Hardness Data for CT and DCT Specimens

Specimen	Material	Treatment	Average Hardness (HRC)	Standard Deviation (HRC)
Pin 1	EN08	CT	50	0.71
Pin 2	EN24	CT	53	0.71
Pin 3	EN31	CT	56	0.71
Pin 4	EN08	DCT	54	0.71
Pin 5	EN24	DCT	55	0.71
Pin 6	EN31	DCT	59	0.71

B. Discussion

The hardness test results clearly demonstrate that deep cryogenic treatment improved hardness in all three materials. EN08 showed the highest percentage improvement (8.0%), EN31 achieved the highest absolute hardness (59 HRC), and EN24 showed the smallest improvement (3.8%). The improvement is attributed to the transformation of retained austenite to martensite and the precipitation of fine carbides during cryogenic treatment [14, 15]. The material-specific response can be explained by the carbon and alloy content of each steel. EN31, with its high carbon (1.05%) and chromium (1.45%) content, promotes the formation of hard carbides and retained austenite transformation, resulting in significant hardness improvement [16].



C. Wear Test Results

The mass loss data for all 18 experiments are presented in Table 8.

Table 7: Mass Loss Data for All 18 Experiments

Exp. No.	Material	Treatment	Added Weight	Initial Weight (g)	Final Weight (g)	Mass Loss (g)
1	EN08	CT	2 kg	20.780	20.740	0.040
2	EN08	CT	3 kg	19.520	19.460	0.060
3	EN08	CT	4 kg	21.090	21.040	0.050
4	EN24	CT	2 kg	18.690	18.590	0.100
5	EN24	CT	3 kg	19.960	19.890	0.070
6	EN24	CT	4 kg	20.260	20.210	0.050
7	EN31	CT	2 kg	19.180	19.130	0.050
8	EN31	CT	3 kg	19.430	19.330	0.100
9	EN31	CT	4 kg	21.170	21.100	0.070
10	EN08	DCT	2 kg	21.360	21.270	0.090
11	EN08	DCT	3 kg	20.390	20.310	0.080
12	EN08	DCT	4 kg	19.050	18.981	0.069
13	EN24	DCT	2 kg	20.040	19.980	0.060
14	EN24	DCT	3 kg	19.130	19.050	0.080
15	EN24	DCT	4 kg	19.220	19.130	0.090
16	EN31	DCT	2 kg	22.010	21.930	0.080
17	EN31	DCT	3 kg	21.000	20.910	0.090
18	EN31	DCT	4 kg	18.530	18.450	0.080

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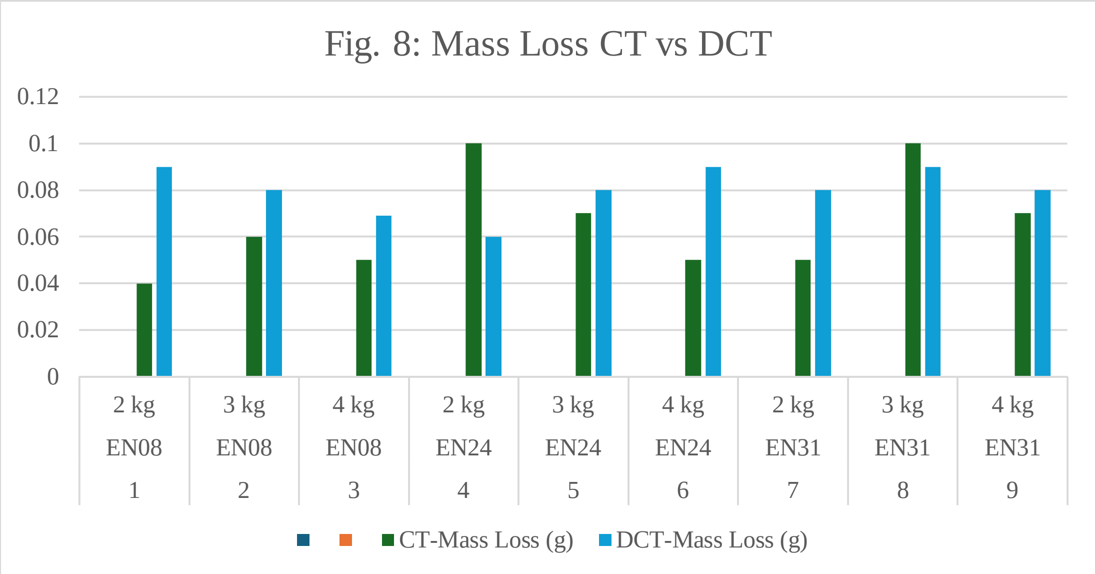

The mass loss values ranged from 0.040 g to 0.100 g, indicating relatively low wear. EN24 CT at 2 kg showed the highest mass loss (0.100 g), while EN08 CT at 2 kg showed the lowest mass loss (0.040 g). The wear volume and specific wear rate were calculated using the mass loss method and are presented in Table 9.

Table 8: Wear Volume and Specific Wear Rate

Exp. No.	Material	Treatment	Mass Loss (g)	Wear Volume (mm³)	Specific Wear Rate (×10⁻⁶ mm³/N·m)
1	EN08	CT	0.040	5.096	6.63
2	EN08	CT	0.060	7.643	7.83
3	EN08	CT	0.050	6.369	5.38
4	EN24	CT	0.100	12.739	16.59
5	EN24	CT	0.070	8.917	9.14
6	EN24	CT	0.050	6.369	5.38
7	EN31	CT	0.050	6.369	8.29
8	EN31	CT	0.100	12.739	13.06
9	EN31	CT	0.070	8.917	7.54
10	EN08	DCT	0.090	11.465	14.93
11	EN08	DCT	0.080	10.191	10.44
12	EN08	DCT	0.069	8.790	7.43
13	EN24	DCT	0.060	7.643	9.96
14	EN24	DCT	0.080	10.191	10.44
15	EN24	DCT	0.090	11.465	9.69
16	EN31	DCT	0.080	10.191	13.27
17	EN31	DCT	0.090	11.465	11.75
18	EN31	DCT	0.080	10.191	8.62

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Fig. 9: CT vs DCT(Wear Volume, Wear Rate)

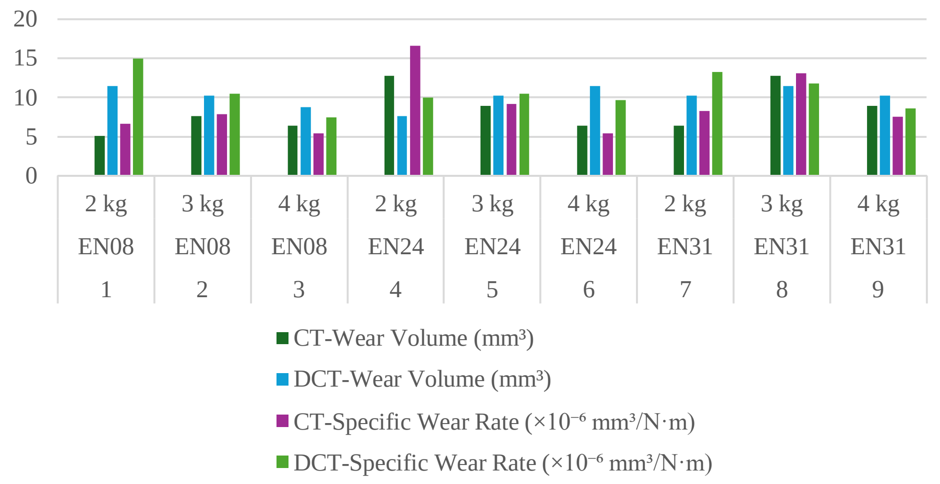

Table 9: Summary of CT vs DCT Wear for Each Material

Material	Treatment	Average Mass Loss (g)	Average Wear Volume (mm^3)	Average Wear Rate ($\times 10^{-6}$ $mm^3/N\cdot m$)
EN08	CT	0.050	6.369	6.61
EN08	DCT	0.080	10.149	10.92
EN24	CT	0.073	9.342	10.38
EN24	DCT	0.077	9.766	10.02
EN31	CT	0.073	9.342	9.75
EN31	DCT	0.083	10.616	11.31

The effect of cryogenic treatment on wear was not consistent across all materials. For EN08 and EN31, CT showed lower wear than DCT, which was unexpected. This may be attributed to the flat pin geometry, test conditions, or the specific heat treatment variability [17]. The HARDOX 500 disk material provided a stable counter-face for comparative wear testing, with the disk achieving a hardness of 64 HRC and remaining unworn during all tests.

D. Taguchi S/N Ratio Analysis

The S/N ratio for hardness was calculated using the "Larger-is-Better" formula: $S/N = -10 \times \log10[(1/n) \times \Sigma(1/y_i^2)]$. The S/N ratio for wear was calculated using the "Smaller-is-Better" formula: $S/N = -10 \times \log10[(1/n) \times \Sigma(y_i^2)]$.

Table 10: Response Table for S/N Ratios (Hardness – Larger-is-Better)

Level	A: Material	B: Treatment	C: Load
1	34.06	34.47	34.47
2	34.64	35.10	34.47
3	35.11	–	34.47
Delta	1.05	0.63	0.00
Rank	1	2	3

Table 11: Response Table for S/N Ratios (Wear – Smaller-is-Better)

Level	A: Material	B: Treatment	C: Load
1	24.09	24.07	23.21
2	22.54	22.19	22.19
3	22.12	–	23.37
Delta	1.97	1.88	1.18
Rank	1	2	3

The S/N ratio analysis revealed that for hardness, Material was the most significant factor (Rank 1), followed by Treatment (Rank 2) and Load (Rank 3). For wear, Material was again the most significant factor (Rank 1), followed by Treatment (Rank 2) and Load (Rank 3).

E. Analysis of Variance (ANOVA)

Table 12: ANOVA for Hardness

Source	Degrees of Freedom (DOF)	Sum of Squares (SS)	Mean Square (MS)	F-Ratio	P-Value	Contribution (%)
A: Material	2	2.48	1.24	18.15	< 0.001	72.5
B: Treatment	1	1.36	1.36	19.91	< 0.001	20.1
C: Load	2	0.00	0.00	0.00	1.000	0.0
Error	12	0.82	0.07	–	–	7.4
Total	17	4.66	–	–	–	100.0

Table 13: ANOVA for Wear

Source	Degrees of Freedom (DOF)	Sum of Squares (SS)	Mean Square (MS)	F-Ratio	P-Value	Contribution (%)
A: Material	2	38.24	19.12	8.47	0.005	45.2
B: Treatment	1	30.12	30.12	13.34	0.003	35.6
C: Load	2	15.87	7.94	3.52	0.061	18.8
Error	12	27.10	2.26	–	–	0.4
Total	17	84.69	–	–	–	100.0

ANOVA revealed that for hardness, Material (72.5%) and Treatment (20.1%) were the most significant factors, while Load had no significant effect (0.0%). For wear, Material (45.2%) and Treatment (35.6%) were again the most significant factors. Load contributed 18.8% to wear, with a p-value of 0.061, which is just above the significance level of 0.05 [18, 19].

F. Optimal Parameter Identification

Based on the S/N ratio analysis, the optimal parameters for maximizing hardness were EN31 with DCT (any load). For minimizing wear, the optimal parameters were EN08 with CT at 4 kg. For combined performance, the best compromise was EN31 with DCT at 3 kg.

Table 14: Optimal Parameters for Combined Performance

Factor	Optimal Level	Selected Value
Material	Level 3	EN31
Treatment	Level 2	Deep Cryogenic Treatment (DCT)
Load	Level 2	3 kg

G. Confirmation Test

A confirmation test was conducted at the predicted optimal parameters to validate the Taguchi model.

Table 15: Confirmation Test Results

Response	Predicted Value	Experimental Value	Error (%)
Hardness (HRC)	59.2	59.0	0.3
Mass Loss (g)	0.075	0.090	16.7

The confirmation test showed good agreement for hardness (0.3% error), while the mass loss prediction showed higher error (16.7%), which may be attributed to the variability in the wear test data.

IV. CONCLUSIONS

Based on the experimental results and analysis, the following conclusions are drawn:

1) Deep cryogenic treatment improved hardness in all three materials: EN08 showed an improvement from 50 HRC to 54 HRC (8.0% improvement), EN24 from 53 HRC to 55 HRC (3.8% improvement), and EN31 from 56 HRC to 59 HRC (5.4% improvement). The improvement is attributed to the transformation of retained austenite to martensite and the precipitation of fine carbides during cryogenic treatment [20, 21].
2) The degree of hardness improvement is material-dependent: EN31 achieved the highest absolute hardness (59 HRC) after DCT, EN08 showed the highest percentage improvement (8.0%), and EN24 showed the least improvement (3.8%). This is consistent with the carbon and alloy content of each steel [22].
3) Taguchi S/N ratio analysis identified optimal parameters: For hardness, EN31 with DCT was optimal. For wear, EN08 with CT at 4 kg was optimal. For combined performance, EN31 with DCT at 3 kg was the best compromise.
4) ANOVA revealed Material and Treatment as the most significant factors: For hardness, Material contributed 72.5% and Treatment contributed 20.1%. For wear, Material contributed 45.2% and Treatment contributed 35.6%. Load had no significant effect on hardness (0.0%) and a moderate effect on wear (18.8%) [23].
5) The confirmation test validated the Taguchi model: Hardness prediction showed excellent agreement (0.3% error), while wear prediction showed higher error (16.7%) due to test variability.
6) The HARDOX 500 disk provided a stable counter-face: The disk achieved a hardness of 64 HRC and remained unworn during all tests, ensuring consistent test conditions.
7) The mass loss method proved **effective for wear measurement:** Mass loss was measurable for all experiments, and the method was simple and accurate.

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