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# FRICTION BEHAVIOUR OF ELECTROLESS Ni-B COATINGS

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## ABSTRACT

This paper presents an experimental study of friction characteristics of electroless Ni–B coatings sliding against steel and optimization of coating process parameters based on the Taguchi method to minimize the same. Experiments are carried out by utilizing the combination of process parameters based on the $L_{27}$ Taguchi orthogonal design with four process parameters, namely, bath temperature, concentration of reducing agent, concentration of nickel source and annealing temperature. It is observed that concentration of reducing agent has the most significant influence in controlling friction characteristics of Ni-B coating. The optimum combination of process parameters for minimum coefficient of friction is obtained from the analysis. The surface morphology and phase content of coatings are also studied with the help of scanning electron microscopy and x-ray diffraction analysis respectively. The wear mechanism is also studied and found to be abrasive in nature.

**Keywords:** Electroless Coating, Ni-B, Taguchi Method, Friction.

## 1. INTRODUCTION

In 1946, Brenner and Riddell [1] discovered a revolutionary coating process, which did not require electricity for its operation and hence was popularized as electroless coating. Electroless plating is an autocatalytic process where the substrate develops a potential when it is dipped in electroless solution called bath that contains a source of metallic ions, reducing agent, complexing agent, stabilizer and other components. Due to the developed potential, both positive and negative ions are attracted towards the substrate surface and release their energy through charge transfer process.

Among the various types of electroless plating, electroless nickel has gained immense popularity due to its ability to provide a hard, wear and corrosion resistant surface [2,3]. Hypophosphite reduced Ni-P coating [4-9] has already been widely accepted and the quest for achieving a superior hard and wear resistant surface has brought Ni-B coatings at the focus of research [10-17]. Aerospace, automotive, chemical and electrical industries utilize electroless nickel–boron plating due to its good solderability, lubricity, high hardness, high wear and abrasion resistance [2].

## 2. TAGUCHI METHOD

Taguchi techniques were developed by G. Taguchi [18]. Taguchi method uses a set of orthogonal arrays to reduce the number of experiments required which may otherwise increase the time and cost of the experiment. Taguchi offers the use of S/N (Signal/Noise) ratio to identify the quality characteristics applied for engineering design problems. The S/N ratio characteristics can be divided into three categories: lower-the-better (LB), higher-the better (HB) and nominal-the best (NB). For the case of minimization of friction, LB characteristic needs to be used. Furthermore, a statistical analysis of variance (ANOVA) [19] is performed to find which process parameters are statistically significant. With the S/N ratio and ANOVA analysis, the optimal combination of coating parameters can be predicted. Finally, a confirmation experiment is conducted to verify the optimal process parameters obtained from the parameter design.

## 3. EXPERIMENTAL DETAILS

### 3.1 Coating Procedure

Mild Steel (AISI 1040) is used as the substrate material (square blocks of size 20 mm × 20 mm × 8 mm) for the deposition of Ni-B films. The substrates are carefully prepared so that their sizes are maintained with precision. The samples are cleaned of any foreign particles and corrosion products prior to coating. Then the samples are cleaned with distilled water. The specimens after thorough cleaning are given a pickling treatment with dilute (18%) hydrochloric acid for one minute to remove any surface layer formed like rust. Finally they are cleaned with distilled water prior to coating. A large number of trial experiments were performed before deciding on the bath composition with the ranges of the coating parameters. Table 1 indicates the bath composition and the operating conditions for successful coating of electroless Ni-B. The cleaned samples were activated in palladium chloride solution at 55°C temperature and placed in the bath for a deposition time of 1 hour. For each sample, the procedure is repeated twice except for the palladium chloride activation in the second round. The range of coating thickness was found to lie between 20-25 microns. After deposition, the samples were taken out of the bath and cleaned using distilled water. Then the samples were heat treated in a box furnace (for 1 h) separately according to the OA. After annealing, the samples were cooled to room temperature (about 25°C) and no artificial cooling is employed. Depending on annealing temperature, it takes 10–15 min time for cooling.

It is important to note that the present study does not consider substrate roughness as the input variable. Thus it is essential that all samples after different stages of processing and prior to coating should have same roughness. But this is extremely difficult to achieve. Thus large numbers of samples are prepared and after all the processing prior to coating these are subjected to roughness evaluation (centre line average, $R_a$). Only those specimens that show insignificant variation (less than 0.1%) in roughness are used for coating deposition.

Table 1: Bath constituents and ranges of values for electroless Ni-B coating

| Parameters | Ranges of parameters |
|---|---|
| Nickel chloride | 15 – 25 g/l |
| Sodium | 0.6 – 1.0 g/l |
| Ethylenediamine | 59 g/l |
| Lead nitrate | 0.0145 g/l |
| Sodium hydroxide | 40 g/l |
| Bath temperature | 85 – 95 °C |
| pH of solution | 12.5 |
| Deposition time | 1 hr |
| Annealing | 250 – 450 °C |

## 3.2 Choice of Process Parameters

There are a large number of factors that can affect the quality of EN coating viz. bath temperature, reducing agent concentration, nickel source concentration, stabilizer concentration, pH of the solution, etc. Based on an intensive literature review it was found that the first three factors (bath temperature, reducing agent concentration and nickel source concentration) were mostly used by the researchers to control EN coating. These three factors were chosen as the main design factors along with their interactions in the present study. The literature review also reports that annealing greatly influences the tribological characteristics of electroless nickel coatings. Hence annealing temperature has been considered as the fourth design factor in this study. Table 2 shows the design factors along with their levels. Three levels, having equal spacing, within the operating range of the parameters were selected for each of the factors. By selecting the three levels, the curvature or non-linearity effects could be studied.

Table 2: Design factors and their levels

| Design Factors | Unit | Levels | | |
|---|---|---|---|---|
| | | 1 | 2 | 3 |
| Bath Temperature (A) | ºC | 85 | $90^a$ | 95 |
| Reducer conc. (B) | (g/l) | 0.6 | $0.8^a$ | 1.0 |
| Nickel source conc. (C) | (g/l) | 15 | $20^a$ | 25 |
| Annealing temp (D) | ºC | 250 | $350^a$ | 450 |

a : initial condition

## 3.3 Response Variable

This study is carried out to consider the friction characteristics of EN coatings as the performance characteristics. The response variable used to accomplish this study is the coefficient of friction (COF). Hence the coating process parameters are optimized with the objective to minimizing the coefficient of friction of electroless Ni-B coatings.

## 3.4 Design of Experiment

The DOE using Taguchi approach can economically satisfy the needs of problem solving and product/process design optimization projects in the manufacturing industry. By learning and applying this technique, engineers, scientists and researchers can significantly reduce the time required for experimental investigations. Based on the Taguchi method, an OA (orthogonal array) is employed to reduce the number of experiments for determining the optimal coating process parameters. An OA provides the shortest possible matrix of combinations in which all the parameters are varied to consider their direct effect as well as interactions simultaneously. Taguchi has tabulated several standard OAs. In this investigation, an $L_{27}$ OA, which has 27 rows corresponding to the number of tests and 26 degrees of freedom (DOFs) with 13 columns at three levels, is chosen. As per the requirements of the $L_{27}$ OA, the 1st column is assigned to bath temperature (A), the 2nd column is assigned to concentration of reducing agent (sodium borohydride) (B), the 5th column is assigned to concentration of source of nickel (nickel chloride) (C), the 9th column is assigned to the annealing temperature (D) while the rest of the columns are assigned to the two-way interactions of the factors and error terms.

## 3.5 Friction Measurement

Friction characteristics of the electroless Ni-B coated specimens are studied under dry, non-lubricated conditions and at ambient temperature of about 28°C and relative humidity of about 85% in a multitribotester apparatus (TR-25, DUCOM, INDIA) using a plate-on-roller configuration. The experiments are conducted with a constant load of 25N and at 60 rpm and for a constant time of 300 s considering the smaller thickness (around 25$\mu$m) of the coatings. The roughness measurement of the coatings prior to the tribological testing is done using a stylus and skid type profilometer, Talysurf (Taylor Hobson, Surtronic 3+). The centre line average roughness values are obtained in the range 0.2–0.8$\mu$m.

### 3.6 Surface Morphology and Compound Analysis

Surface morphology of the EN coatings is studied by SEM (JEOL, JSM-6360) in order to analyse the microstructure of the deposited coatings before and after annealing at different heat treatment temperatures to see the effect of annealing temperature. SEM is also done after the tribological testing to see the sliding track patterns. The different precipitated phases of the deposits both before and after annealing were analyzed by X-ray diffraction (XRD) analyzer (Rigaku, Ultima III).

## 4. RESULTS AND DISCUSSION

### 4.1 Analysis of Signal to Noise Ratio

As an evaluation tool for determining robustness, the signal-to-noise (S/N) ratio is preferred to simple averages of results as the former can capture the variability of the results within a trial condition. Hence Taguchi technique utilizes the S/N ratio approach to measure the quality characteristic deviating from the desired value. In the present work S/N ratio analysis is done with coefficient of friction as the performance index. The S/N ratio for coefficient of friction is calculated using LB (Lower the better) criterion and is given by

$$S/N = -10\log(\sum y^2/n) \quad (1)$$

where y is the observed data and n is the number of observations. Table 3 shows the experimental COF values along with their S/N ratios. As the experimental design is orthogonal, it is possible to separate out the effect of each coating parameter at different levels. The mean S/N for each level of the factors A, B, C and D is summarized in Table 4. All the calculations are performed using Minitab® [20]. The response table includes ranks based on Delta statistics, which compare the relative magnitude of effects. The Delta statistic is the highest average for each factor minus the lowest average for the same. Ranks are assigned based on Delta values; rank 1 is assigned to the highest Delta value, rank 2 to the second highest Delta value, and so on. The corresponding main effects and interaction effects plots between the process parameters are also shown in Fig 1 and Fig 2 respectively. In the main effects plot if the line for particular parameter is near horizontal, then the parameter has no significant effect. On the other hand, a parameter for which the line has the highest inclination will have the most significant effect. From the main effects plot (Fig 1) it is clear that factor B i.e. the concentration of the reducing agent is the most significant factor while factor A i.e. bath temperature is moderately significant. In case of interaction plots non-parallelism of the parameter effects are observed. If the lines on the interaction plots are non-parallel, interaction occurs and if the lines cross, strong interactions occur between parameters. From the interaction plots (Fig 2) it can be observed that the interaction between A and C and that between B and C are somewhat significant as far as the friction characteristics are concerned. Thus from the present analysis it is clear that the concentration of reducing agent (B) is the most influencing parameter for COF of electroless Ni-B coatings. The optimal process parameter combination is the one that yields maximum mean S/N ratio i.e. minimum COF and is found to be A1B1C1D3.

Table 3: Experimental results along with S/N ratio

| **Exp. No.** | **COF** | **S/N Ratio** |
|---|---|---|
| 1 | 0.184 | 14.72728 |
| 2 | 0.107 | 19.41232 |
| 3 | 0.132 | 17.58852 |
| 4 | 0.314 | 10.06141 |
| 5 | 0.411 | 7.723164 |
| 6 | 0.108 | 19.33152 |
| 7 | 0.300 | 10.45757 |
| 8 | 0.131 | 17.65457 |
| 9 | 0.571 | 4.867278 |
| 10 | 0.093 | 20.63034 |
| 11 | 0.282 | 10.99502 |
| 12 | 0.209 | 13.59707 |
| 13 | 0.083 | 21.61844 |
| 14 | 0.246 | 12.1813 |
| 15 | 0.311 | 10.14479 |
| 16 | 0.368 | 8.683044 |
| 17 | 0.326 | 9.735648 |
| 18 | 0.310 | 10.17277 |
| 19 | 0.207 | 13.68059 |
| 20 | 0.449 | 6.955073 |
| 21 | 0.274 | 11.24499 |
| 22 | 0.409 | 7.765534 |
| 23 | 0.124 | 18.13157 |
| 24 | 0.267 | 11.46977 |
| 25 | 0.364 | 8.777972 |
| 26 | 0.255 | 11.8692 |
| 27 | 0.477 | 6.429632 |

Table 4: Response table for mean S/N ratio

| **Level** | **A** | **B** | **C** | **D** |
|---|---|---|---|---|
| 1 | 13.536 | 14.315 | 12.934 | 11.925 |
| 2 | 13.084 | 13.159 | 12.740 | 12.556 |
| 3 | 10.703 | 9.850 | 11.650 | 12.842 |
| Delta | 2.833 | 4.465 | 1.284 | 0.917 |
| Rank | 2 | 1 | 3 | 4 |

Total mean S/N Ratio = 12.441 dB

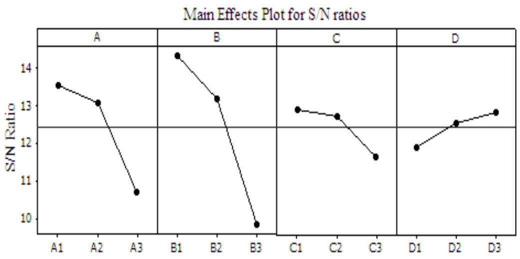

Fig 1. Main effects plot for mean S/N ratio

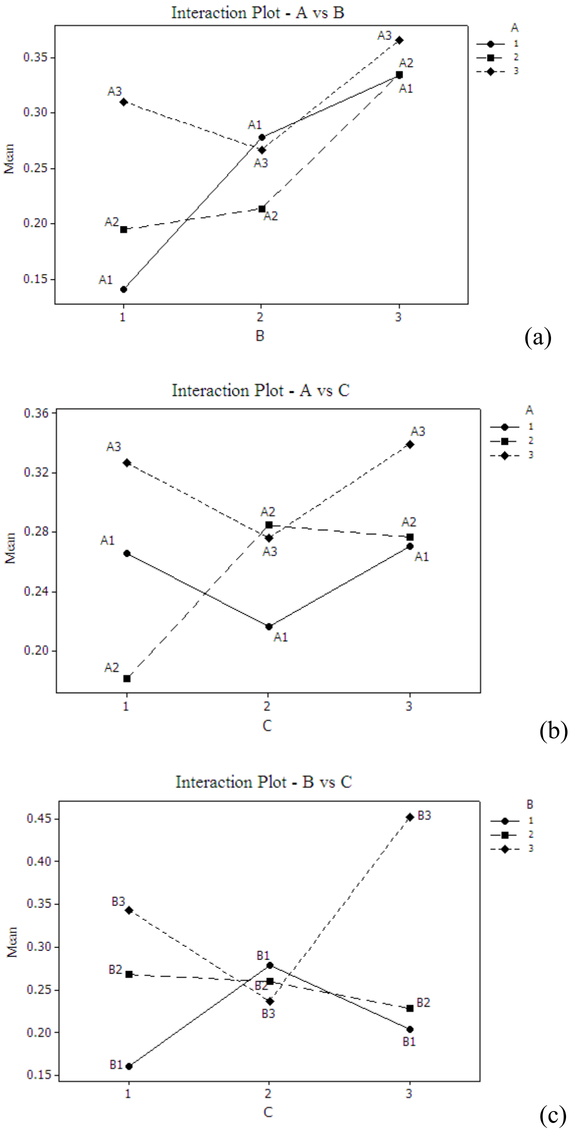

Fig 2. Interaction effects plot for mean COF (a) A versus B, (b) A versus C and (c) B versus C.

## 4.2 Analysis of Variance (ANOVA)

The purpose of the ANOVA is to investigate which of the process parameters significantly affect the performance characteristics. This is accomplished by separating the total variability of the S/N ratios, which is measured by the sum of the squared deviations from the total mean of the S/N ratio, into contributions by each of the process parameters and the error. In the present study ANOVA is performed using Minitab® [20]. ANOVA results for COF of electroless Ni-B coating is shown in Table 5. In ANOVA a ratio called F-ratio, which is the ratio between the regression mean square and mean square error is used to measure the significance of the parameters under investigation with respect to the variance of all the terms included in the error term at the desired significance level, α. A calculated F-ratio which is higher than the tabulated F-ratio indicates that the factor is significant at desired α level. ANOVA table also shows the percentage contribution of each parameter. It is seen that parameter B, i.e. concentration of reducing agent has got the most significant influence on coefficient of friction at the confidence level of 65% within the specific test range. No other parameter is significant at the said level of confidence. Also none of the interaction is found to have significant effect on the friction characteristics of electroless Ni-B coating but interaction between bath temperature and concentration of nickel source (A × C) and that between concentration of reducing agent and concentration of nickel source (B × C) have got somewhat contribution in controlling the friction coefficient.

Table 5: ANOVA table for coefficient of friction

| **Source** | **DF** | **SS** | **MS** | **F** | **% contribution** |
|---|---|---|---|---|---|
| A | 2 | 41.71 | 20.85 | 0.57 | 7.33 |
| B | 2 | 96.66 | 48.33 | 1.33* | 17.00 |
| C | 2 | 8.62 | 4.31 | 0.12 | 1.52 |
| D | 2 | 3.96 | 1.98 | 0.05 | 0.70 |
| A*B | 4 | 42.80 | 10.70 | 0.29 | 7.52 |
| A*C | 4 | 87.56 | 21.89 | 0.60 | 15.39 |
| B*C | 4 | 69.72 | 17.43 | 0.48 | 12.25 |
| Error | 6 | 217.94 | 36.32 |  | 38.30 |
| Total | 26 | 568.97 |  |  | 100 |

* - significant parameters and interactions
($F_{0.35,\,2,\,6} = 1.26$)

## 4.3 Confirmation Test

Once the optimal level of the process parameters is selected, the final step is to predict and verify the improvement of the performance characteristic using the optimal level of the process parameters. The estimated S/N ratio $\hat{\gamma}$ using the optimal level of the process parameters can be calculated as

$$\hat{\gamma} = \gamma_m + \sum_{i=1}^{o} \left( \bar{\gamma}_i - \gamma_m \right) \quad (2)$$

where $\gamma_m$ is the total mean S/N ratio, $\bar{\gamma}_i$ is the mean S/N ratio at the optimal level, and o is the number of the main design parameters that significantly affect the COF of electroless Ni-B coating. Table 6 shows the comparison of the estimated S/N ratio with the actual S/N ratio using the optimal parameters. The increase of the S/N ratio from the initial coating parameters to the optimal coating parameters is 9.742 dB which means friction is reduced by about 67%.

Table 6: Result of the confirmation experiment for friction coefficient

| | Initial parameters | Optimal Parameters | Experiment |
|---|---|---|---|
| Level | A2B2C2D2 | A1B1C1D3 | A1B1C1D3 |
| COF | 0.396 | | 0.129 |
| S/N ratio (dB) | 8.046 | 12.441 | 17.788 |

Improvement of S/N ratio = 9.742 dB

### 4.4 Surface Morphology and Phase Structure

Surface morphology study of the coatings is done by SEM in order to analyse the effect of heat treatment on the microstructure of the coatings for some of the samples at random and they show similar qualitative change in microstructure. Fig 3 shows the SEM micrographs of the samples in as deposited and under heat treated conditions. It is seen that the electroless Ni-B coatings in general exhibit a defect free surface with distribution of Ni-B nodules, more like that of a cauliflower surface which indicates that the coating possesses a lubricious behaviour. The surface of the coating appears dense and light grey in colour with low porosity. When heat treated, the Ni-B nodules grow in size giving rise to a coarse-grained structure. This indicates that in as deposited condition the structure is a mixture of amorphous and microcrystalline which becomes crystalline with heat treatment. This is further supported by the XRD patterns of Ni-B deposits in as deposited and heat treated condition (Fig 4). The XRD patterns in as deposited condition is a collection of microcrystalline peaks. But with heat treatment at 450°C for one hour, broad peaks of Ni, $Ni_2B$ and $Ni_3B$ are produced.

SEM micrograph of the worn surface is shown in Fig 5. The presence of longitudinal grooves along the sliding direction with high degree of plasticity can be clearly observed. This is indicative of the occurrence of micro-cutting and micro-ploughing effect and characterized as ductile failure. Almost no pits or prows are observed on the worn surface. Hence it can be concluded that the abrasive wear is the predominant phenomenon. The same trend is observed for other combinations of deposition parameters within the experimental regime considered in this study.

The influence of four factors, viz., bath temperature, concentration of nickel source, concentration of reducing agent and annealing temperature is considered in the present study. Future study may attempt to evaluate the influence of other factors viz. concentration of stabilizer, pH of solution, substrate roughness, etc on the friction characteristics of electroless Ni-B coating.

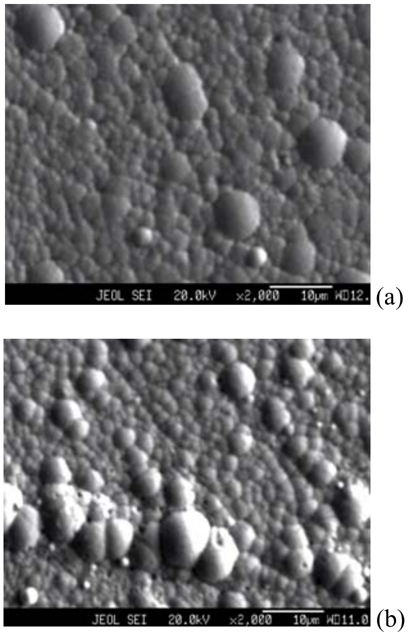

Fig 3. SEM micrographs of the coating surfaces: (a) as-deposited and (d) heat treated at 450ºC.

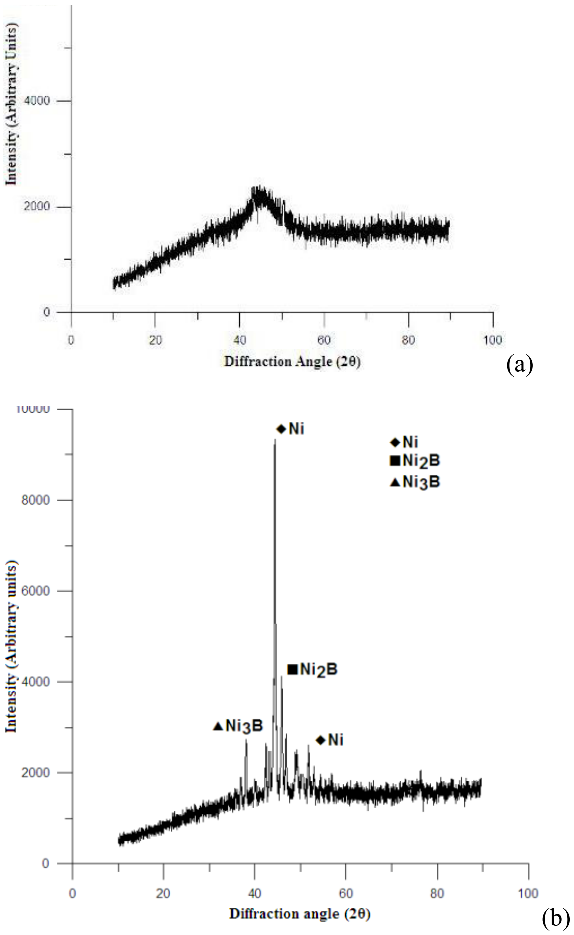

Fig 4. XRD patterns of electroless Ni–B deposit in

(a) as-deposited and (b) heat treated at 450ºC

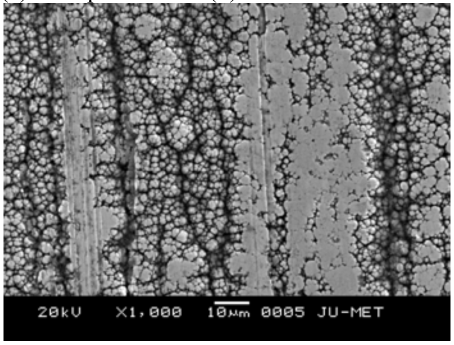

Fig 5. SEM micrograph of coating surface after friction testing

## 5. CONCLUSION

In the present study Taguchi orthogonal array is used to optimize the coating process parameters (bath temperature, concentration of reducing agent and concentration of nickel source) together with annealing temperature in order to minimize coefficient of friction of electroless Ni-B coating. The following conclusions can be drawn:

- Concentration of reducing agent (B) is the most important parameter that significantly affects the coefficient of friction at a confidence level of 65%.
- Interaction between bath temperature and concentration of nickel source (A × C) and that between concentration of reducing agent and concentration of nickel source (B × C) have got somewhat contribution in controlling the friction coefficient.
- The optimal parameter combination for minimum friction is A1B1C1D3.
- The coefficient of friction is reduced by about 67% at the optimal condition compared to the initial condition.

The microstructural study through SEM micrographs reveals that the coating has a cauliflower like structure with no obvious surface damage and low porosity. XRD analysis shows that the coating is in general amorphous in as deposited condition and turns crystalline with heat treatment. The sliding tracks show that abrasive failure is the predominant phenomenon.

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