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**Study of changes in Wear Resistance of Cr-V Ledeburitic Tool  
Steel Due to Cryogenic Processing**

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## Introduction

Chromium-vanadium (Cr-V) ledeburitic steels are commonly used in numerous industrial processes and operations. These materials must function in harsh environments to withstand wear and plastic deformation, and they must also meet industrial standards for high production stability and reliability. However, these steels should exhibit acceptable level of fracture toughness. The main alloying element in Cr-V ledeburitic steel is chromium. Different kinds of more or less-stable carbides are formed by it. The second typical component of Cr-V ledeburitic steels is vanadium. Vanadium forms extremely stable MC-carbides because it has a strong affinity for carbon. The small size and high thermal stability of MC-carbide particles make vanadium-containing steels resistant to grain coarsening during austenitizing. MC-carbide particles preserve the relatively favorable mechanical properties after the heat treatment [1], [2].

Every year, a growing need for energy efficiency, cost cutting, and improved product performance is referenced as substantiation for the development of new materials and processes for the automotive, aerospace, biomedical industries, and others [3-6]. A number of tactics are currently being developed to meet these demands, and one technique that is frequently used to enhance the properties of metallic materials is heat treatment. A common process called "heat treatment" involves repeatedly heating and cooling a material to a predetermined temperature for a predetermined amount of time in order to modify the target material in a purposeful manner. Conventional heat treatment, which keeps processing temperatures above zero degrees (°C), is the most widely used heat treatment technique. But over the past century, many scientists have experimented with cryogenic or sub-zero treatment. Metallic materials were cooled to temperatures below zero [7] in order to enhance the treated material's wear resistance. Later developments of the technique claim that a number of additional properties, such as strength, hardness, toughness, fracture toughness, and corrosion resistance, have been added to the list of properties that can be altered by cryogenic treatment.

Many studies have been conducted in the past to better understand the behaviour of Cr-V ledeburitic steels under various conditions, such as austenitizing temperatures and times, cryogenic treatment without tempering, the impact of cryogenic treatment on the microstructure, wear resistance, and fracture toughness, and the response to tempering after both short- and long-term cryogenic treatment.

Initial investigations into the microstructure of Vanadis 6 cold work tool steel reveal the presence of  $M_7C_3$ - and MC-carbides. The same material was subjected to different austenitizing temperatures ranging from 1000 to 1200 °C. According to the results,  $M_7C_3$ -carbides are less stable than MC-carbides. The  $M_7C_3$ -carbides subsequently underwent extensive dissolution in the austenite and were not found above 1100 °C. The MC-carbides, on the other hand, were essentially unaffected by this change and only began to dissolve at a temperature of 1200 °C. The highest hardness of 65.2 HRC was observed at a temperature of 1025 °C [2].

In order to further investigate the microstructure, phase constitution, and hardness of the Vanadis 6 cold work tool steel, cryogenic treatment was applied for 4 hours at a temperature of -196 °C. The results show that compared to conventional heat-treatment (CHT), the introduction of cryogenic treatment results in a more complete martensitic transformation. Compared to material that has undergone conventional heat treatment, the microstructure of steel that has undergone cryogen treatment contains a greater amount of small globular carbides. The cryogenic treatment has resulted in a modest increase in the material's bulk hardness. Because the retained austenite content is reduced more significantly when the treatment is done at a higher austenitizing temperature, the increase in hardness is more noticeable [2].

To continue the investigation, Vanadis 6 ledeburitic steel was conventionally quenched and cryotreated for 4, 10, and 17 hours, respectively, at temperatures of -90 and -196 °C. The obtained results designate that i) cryogenic treatment encourage time-dependent reduction of retained austenite amount, ii) enhanced population density of small globular carbides after cryogenic treatments; the population density is directly proportioned to the duration of cryogenic treatment, iii) application of cryogenic treatment results in overall refinement of the microstructure, iv) decomposition of retained austenite is accelerated by cryogenic treatments, v) tempering reduces the density of small globular carbides, vi) cryogenic treatments modifies the precipitation behaviour of carbides; vii) the application of cryogenic treatment results in the complete loss of the secondary hardening peak, viii) the overall tempering response of the steel is a result of competition between the tempering of the martensite, decomposition of the retained austenite, deviation in the small globular carbides count, and precipitation of carbides; higher hardness of cryogenically treated and low-temperature tempered steel can be attributed to lower retained austenite amount, and much higher number of small globular carbides [8], [9]. The cryogenic treatment of the material not only reduces or eliminates the retained austenite content but also it considerably modifies the precipitation behaviour of the carbides resulting into improved hardness and homogeneity of the structure [9], [10].

# 1 Objectives of the Thesis

In the present PhD thesis, the tribological performance of Cr-V ledeburitic steel Vanadis 6 is investigated. The examined steel was subjected to different heat treatment strategies whereas: The austenitizing temperature was kept constant and the cryogenic treatments were carried out at -75, -140 or -196 °C, each for 17 h. Each cryogenic treatment was followed by either low- (170 °C) or high-temperature (530 °C) tempering.

The objective of this study is to find the optimum process parameters in order to obtain the best wear performance of Vanadis 6 steel. For this reason, the influence of input variables such as cryogenic temperature, tempering temperature, sliding velocity, and load conditions are studied on the output response such as coefficient of friction, wear rate, counterpart material transfer. Because the goal of the research is to better understand abrasive, adhesive-abrasive, and adhesive wear mechanisms, besides the standard counterparts materials such as Al<sub>2</sub>O<sub>3</sub>, 100Cr6, also CuSn6 bronze was chosen. The fundamental reason for using CuSn6 is because of its low shear strength, which allows only adhesive wear to predominate. The design of experiments methodology was used to identify the optimum conditions. The details of the methodology, and experiments are elaborated in further sections.

The following are the selective goals of the thesis, chosen in accordance with the main objectives:

- To determine the main microstructural changes that occur in the examined steel due to cryogenic treatments.
- To realize comprehensive research of the wear performance of the examined steel, by using a pin-on-disc apparatus.
- To find the optimum conditions of processing with respect to maximize the wear performance, minimize the friction coefficient and reduce the counterpart's material transfer.
- To determine the main wear mechanisms.

## 2 Experimental and Tribological Studies

### 2.1 Experimental procedure

The conventional heat treatment of Cr-V ledeburitic tool steel was performed in a vacuum furnace for 30 minutes at 1050°C, followed by gas quenching. After that the samples are divided into six batches to perform cryogenic treatment. The cryogenic treatment was performed at different temperatures including -75, -140, and -196°C for the duration of 17 hours. Later, the samples are divided into two batches, with the first batch being tempered at 170°C and the later batch was tempered at 530°C for the duration of 2 hours. **Figure 1** displays a detailed description of the heat treatment schedules used in the investigations.

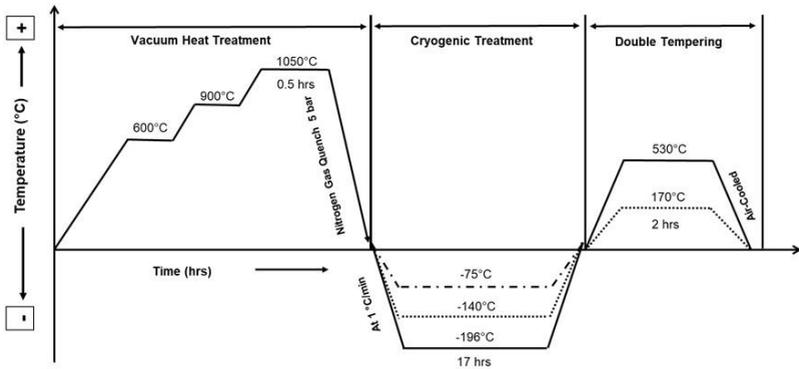


Figure 1. Schematic representation of process parameters used in heat treatment.

The microstructural features were determined by means of the scanning electron microscopy (SEM) using a JEOL JSM-7600F operating at the acceleration voltage of 15 kV in regimes of secondary electrons (SE) and back-scattered electrons (BE). The microscope was equipped with an Oxford Instruments X-max 50 spectrometer for energy-dispersive X-ray spectroscopy (EDX) operated by INCA software. According to the ASTM E384-17 [11] standard, the hardness measurements were carried out on flat, mirror-polished samples using a calibrated Vickers hardness tester. A load of 10 kgf (98N) is used during the indentation process and minimum of five readings were obtained to estimate the average hardness of individual sample. Moreover, the bulk hardness of the samples was evaluated using the ASTM E18 [12] standard procedure and these tests were performed on verified and calibrated mechanical Rockwell hardness tester using Rockwell C scale with an applied load of 150 kg and dwell time of 10 s. Five readings were collected at various places to estimate the average hardness value for each sample.

Flat samples with measurements of 30 x 30 x 6 mm were used for tribological tests. Three different counterparts were used on a tribometer made by CSM to conduct wear tests on mirror polished samples in accordance with ASTM G99-17 standard [13]. First, the

experimental material was tested against a hard alumina ( $\text{Al}_2\text{O}_3$ ) counterpart to simulate abrasive wear performance. Next, a standard bearing steel (100Cr6) counterpart was used to simulate mixed wear conditions of adhesive and abrasive. Finally, a soft bronze (CuSn6) counterpart was used to simulate adhesive wear. The design of experiments methodology is a powerful tool for data collection and evaluation that can be used in a variety of process optimization studies. Therefore, Taguchi method was chosen as an appropriate method to study the tribological performance of the experimental material as a single parameter optimization technique [14, [15] and hybrid Taguchi and Grey relation analysis was chosen as an appropriate method for multi-optimization technique [16], [17]. MINITAB software was used for statistical data analysis. To describe the worn surfaces and identify wear mechanisms, a scanning electron microscope and EDS spectroscopy were used.

## **2.2 Results and discussion**

### **2.2.1 Microstructure**

The microstructures of Cr-V ledeburitic tool steel following various cryogenic treatments at  $-75$ ,  $-140$ , and  $-196^\circ\text{C}$ , for 17 hrs, and after performing tempering treatment at  $170^\circ\text{C}$  and  $530^\circ\text{C}$  is shown in **Figure 2**. Observations show that steel contains carbides and matrix regardless of the heat treatment schedules used. The matrix is martensitic with some amount of retained austenite, observed in-between the martensite domains. Eutectic carbides (ECs), secondary carbides (SCs), and small globular carbides (SGCs) are the three distinct carbides that can be seen in the microstructures. In the scientific literature, there is a clear understanding of the origin and nature of these carbides [1], [2], [8], [9]. For instance, MC-type eutectic carbides are vanadium-rich and have a typical size of roughly  $1\ \mu\text{m}$ .  $\text{M}_7\text{C}_3$ -type secondary carbides, on the other hand, are chromium-rich and typically range in size from  $1$ - $3\ \mu\text{m}$ . Finally, the SGCs are cementite carbides with sizes averaging  $300$  nanometers [18].

Since the hardening parameters remained constant for various treatments, it should be noted that recent reports [2], [8] claim that cryogenic treatment does not change the amounts or population densities of eutectic and secondary carbides. The reason for this is that these carbides are stable at much higher temperatures, so neither the cryogenic treatment nor the tempering affect their quantitative characteristics [2], [8], [19]. On the other hand, cryogenic treatments change the number and population density of SGCs. The population densities of SGCs for various cryogenically treated and tempered specimens gathered from recent studies [2], [8], [18] are shown in **Figure 3**. Cryogenic treatment at  $140^\circ\text{C}$  produced the highest population density of SGCs; refer **Figure 3b**. Recently, Jurci et al. acknowledged that these small globular carbides are cementite, a transient and metastable carbide phase in the relevant system, and that tempering treatment reduces their volume and population density [20].

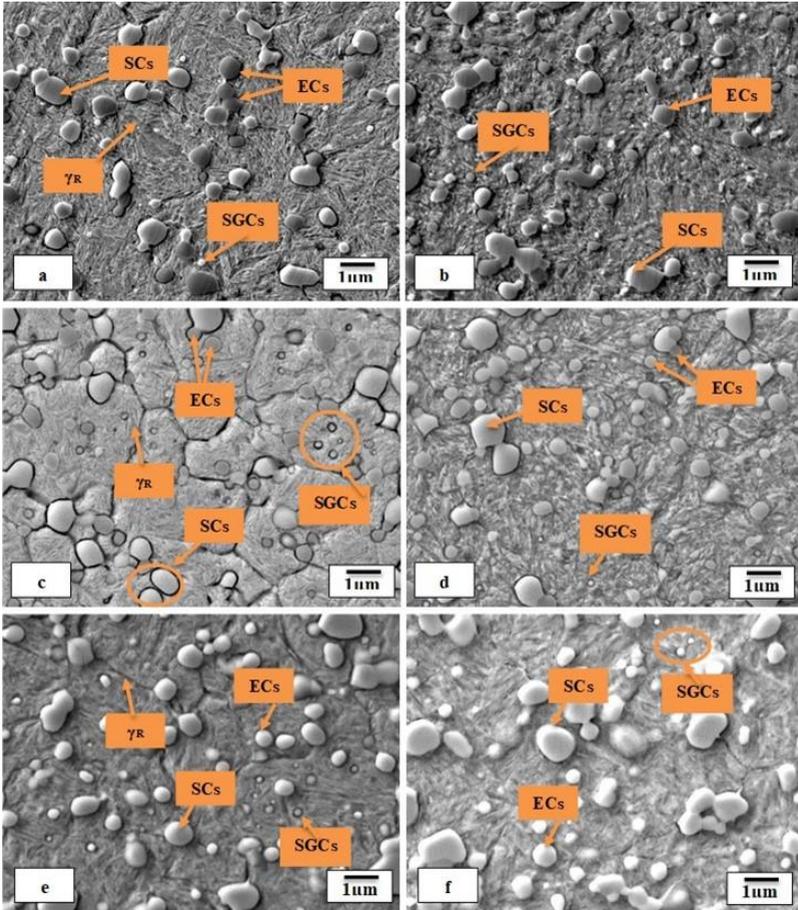


Figure 2. SEM micrographs of cryo-treated specimens: (a)  $-75\text{ }^{\circ}\text{C}$ , tempered at  $170\text{ }^{\circ}\text{C}$ , (b)  $-75\text{ }^{\circ}\text{C}$ , tempered at  $530\text{ }^{\circ}\text{C}$ , (c)  $-140\text{ }^{\circ}\text{C}$ , tempered at  $170\text{ }^{\circ}\text{C}$ , (d)  $-140\text{ }^{\circ}\text{C}$ , tempered at  $530\text{ }^{\circ}\text{C}$ , (e)  $-196\text{ }^{\circ}\text{C}$ , tempered at  $170\text{ }^{\circ}\text{C}$ , (f)  $-196\text{ }^{\circ}\text{C}$ , tempered at  $530\text{ }^{\circ}\text{C}$

Another significant benefit of cryogenic treatment over conventional treatment is the reduced amount of retained austenite (see **Figure 3a**). For instance, when Cr-V ledeburitic steel was conventionally heat treated, it was reported that 20% of the austenite was retained [2], [8]. The obtained results demonstrate that cryogenic treatment reduces the amount of retained austenite by more than 65% when compared to conventional heat treatment. The volumes of retained austenite for Cr-V ledeburitic steel treated at  $-75$ ,  $-140$ , and  $-196\text{ }^{\circ}\text{C}$ , respectively, were found to be 5.5, 4.3, and 2.1% when tempered at  $170\text{ }^{\circ}\text{C}$  [2], [8], [18]. It is important to note that the amount of retained austenite is most effectively reduced by cryogenic treatment at the lowest temperature used ( $-196\text{ }^{\circ}\text{C}$ ). On the other hand, when tempering at  $530\text{ }^{\circ}\text{C}$ , the retained austenite is completely removed from all specimens.

**Figure 4** shows the X-ray diffraction profiles of specimens that were both conventionally treated and cryotreated at  $-140\text{ }^{\circ}\text{C}$  for different durations, obtained from the literature [18]. The diffraction peaks of martensite, retained austenite, and various carbides are visible in the XRD profiles. When a specimen is conventionally treated, the peaks of retained austenite at  $(111)\gamma$ ,  $(200)\gamma$ ,  $(220)\gamma$ , and  $(311)\gamma$  are clearly visible in the diffraction profile. However, after cryogenic treatments, the last two peaks almost completely vanish from the XRD profiles. Additionally, cryogenic treatment significantly lowers the retained austenite as evidenced by the fact that the height of both the  $(111)\gamma$  and  $(200)\gamma$  peaks is significantly reduced after treatment.

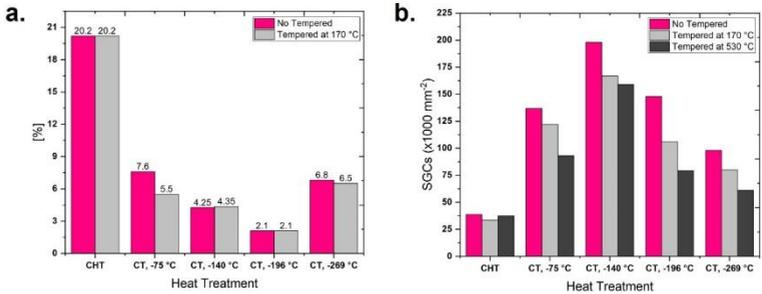


Figure 3. Amounts of retained austenite (a) and small globular carbides (b) at different treatments for Vanadis 6 steel [2], [8], [18].

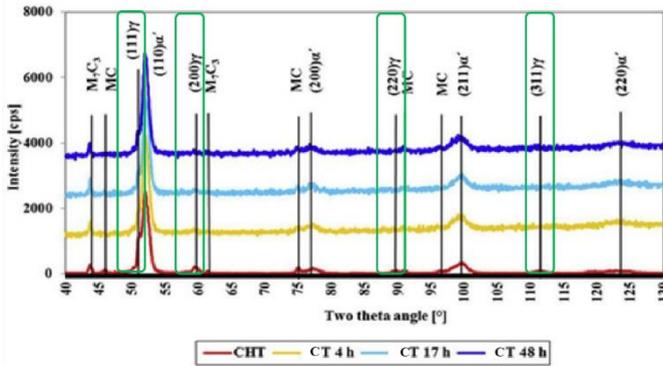


Figure 4. X-ray diffraction profiles of conventional and cryotreated (CT) specimens at different durations [18].

### 2.2.2 Hardness behavior

The mean Vickers and Rockwell hardness values of differently processed specimens are shown in **Figure 5**. According to the measured hardness values, the specimens can generally be divided into two groups. Regardless of the temperature of

cryogenic treatment, tempering at 170 °C results in much higher hardness (by about 190 HV10) than tempering at 530 °C, and cryogenic treatment at -140 °C obtained the highest hardness of 905 HV compared to other treatments. The principle explanation is that the matrix is primarily made of martensite, which gradually softens as tempering temperature rises. Additionally, one can anticipate a small but certain contribution from additional carbides. Due to the higher carbide counts following low-temperature tempering, their contribution is inevitably greater than in the state following high-temperature tempering. An opposite tendency is evident in the  $\gamma_R$  amounts. High-temperature tempering completely eliminates this (soft) phase, which counteracts both martensite softening and decreases in carbide count. However, the above-mentioned martensite softening and decrease in carbide counts cannot be balanced by this counteraction. At constant tempering temperatures, the effect of various cryogenic treatments on the final steel macro-hardness can be regarded as minimal.

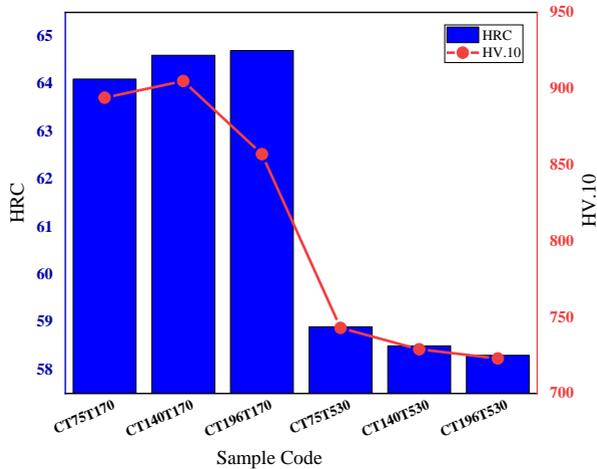


Figure 5. Vickers hardness vs Rockwell hardness of different cryo-treated and tempered specimens.

### 2.2.3 Design of experiments

Pin-on-disk dry sliding experiments against all three counterparts ( $Al_2O_3$ , 100Cr6, CuSn6) were designed, planned, and conducted based on the run order generated by the Taguchi mixed level ( $2^1 3^3$ ) design using the L18 orthogonal array. The details of the selected control factors and their corresponding levels are presented in **Table 1**. The experimental results of individual counterparts will be discussed separately in the following sections. For all of the planned designs, statistical analyses were conducted using MINITAB 19.

Table 1. Controlled factors and levels used in the design of experiments.

Factor levels	Factors			
	CT [°C]	TT [°C]	SV [m/s]	L [N]
1	-75	170	0.064	1
2	-140	530	0.128	5
3	-196	-	0.1885	10

#### 2.2.4 Alumina counterpart ( $Al_2O_3$ )

As the counterpart in the ball on flat configuration, commercially available alumina balls with a 6 mm diameter were chosen. The friction coefficient (FC) and wear rate ( $W_R$ ) are response variables that are taken into account because it is expected that the harder counterpart will experience abrasive wear.

The experimental results for FC and  $W_R$  are shown in **Table 2**. The standard deviation for the FC and  $W_R$ , respectively, in the provided data is 0.06 and 1.001. The transformation technique was used to reduce the standard deviation and enhance the normality of the data because the standard deviation of wear rate data is on the higher side.

Table 2. Experimental results for FC and  $W_R$  [ $Al_2O_3$ ].

Exp.no	CT [°C]	TT [°C]	SV [m/s]	L [N]	FC	WR [mm <sup>3</sup> /m×10 <sup>-7</sup> ]
1	-75	170	0.064	1	0.820	0.472
2	-75	170	0.128	5	0.712	0.967
3	-75	170	0.1885	10	0.637	2.014
4	-140	170	0.064	1	0.833	0.278
5	-140	170	0.128	5	0.713	1.063
6	-140	170	0.1885	10	0.613	1.579
7	-196	170	0.064	5	0.696	0.330
8	-196	170	0.128	10	0.660	1.902
9	-196	170	0.1885	1	0.770	0.463
10	-75	530	0.064	10	0.753	2.972
11	-75	530	0.128	1	0.840	0.553
12	-75	530	0.1885	5	0.748	1.476
13	-140	530	0.064	5	0.708	1.186
14	-140	530	0.128	10	0.749	3.224
15	-140	530	0.1885	1	0.776	0.363
16	-196	530	0.064	10	0.776	3.226
17	-196	530	0.128	1	0.815	0.445
18	-196	530	0.1885	5	0.696	1.713

The objective of the study is to reduce FC and  $W_R$ . As a result, they are consequently examined in Minitab 19 using the lower-the-better quality characteristic. **Table 3** shows the original and transformed data as well as the corresponding signal to noise ratios (SN ratios) for friction coefficient (FC) and wear rate ( $W_R$ ).

Table 3. Experimental results after transformation for FC and  $W_R$  [ $Al_2O_3$ ].

Exp.no	CT	TT	SV	L	FC	SN ratio	WR	SN ratio
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1	-75	170	0.064	1	0.820	1.724	0.472	0.456
2	-75	170	0.128	5	0.712	2.950	0.967	0.020
3	-75	170	0.1885	10	0.637	3.917	2.014	-0.426
4	-140	170	0.064	1	0.833	1.587	0.278	0.778
5	-140	170	0.128	5	0.713	2.938	1.063	-0.037
6	-140	170	0.1885	10	0.613	4.251	1.579	-0.278
7	-196	170	0.064	5	0.696	3.148	0.330	0.673
8	-196	170	0.128	10	0.660	3.609	1.902	-0.391
9	-196	170	0.1885	1	0.770	2.270	0.463	0.468
10	-75	530	0.064	10	0.753	2.464	2.972	-0.662
11	-75	530	0.128	1	0.840	1.514	0.553	0.360
12	-75	530	0.1885	5	0.748	2.522	1.476	-0.237
13	-140	530	0.064	5	0.708	2.999	1.186	-0.104
14	-140	530	0.128	10	0.749	2.510	3.224	-0.712
15	-140	530	0.1885	1	0.776	2.203	0.363	0.616
16	-196	530	0.064	10	0.776	2.203	3.226	-0.712
17	-196	530	0.128	1	0.815	1.777	0.445	0.493
18	-196	530	0.1885	5	0.696	3.148	1.713	-0.327

The main effects plot in Taguchi analysis was used to establish the correlations in relation to various factors. The main effects plot for the SN ratios of friction coefficient can be seen in **Figure 6**.

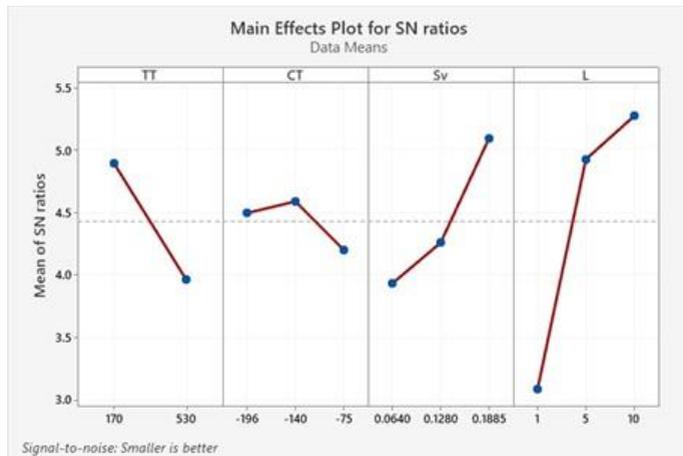


Figure 6. Main effects plot for SN ratios, FC  $[Al_2O_3]$ .

The FC value increases with an increase in tempering temperature, according to the main effects plot. In contrast, cryogenic treatment has little effect on the FC; however, samples treated at  $-140^{\circ}C$  have less FC than samples treated at other cryogenic temperatures. The sliding velocity has a significant impact on FC value; for example, higher sliding velocity results in lower FC, and vice versa. The load factor has the greatest impact on the FC of all the other factors. For example, higher loads can result in lower FC values,

and vice versa. The ANOVA study furthermore establishes the statistical significance and individual factor contribution to the FC, as shown in **Table 4**. The most significant factor affecting the FC according to the ANOVA table is load, which accounts for 56.75% of the FC, followed by sliding velocity (14.75%), tempering temperature (13.47%), and cryogenic treatment (1.68%) (which has no statistical significance). The  $R^2$  value for ANOVA model is 86.66 % and the  $R^2$  (adj) value is 77.32 %. In other words, this model explains 86.66% of the total variation in the results of the experiments. The close difference and higher  $R^2$  and adj  $R^2$  values imply that the model proposed is accurate.

Table 4. ANOVA table for FC [ $Al_2O_3$ ].

Source	DF	Seq SS	Adj SS	Adj MS	F	P	% C
TT	1	1.419	1.419	1.419	10.10	0.010	13.47
CT	2	0.177	0.177	0.088	0.63	0.552	1.68
SV	2	1.553	1.553	0.776	5.53	0.024	14.75
L	2	5.978	5.978	2.989	21.27	0.000	56.75
Residual Error	10	1.405	1.405	0.140			
Total	17	10.533					

In **Figure 7**, the main effects plot for the SN ratios of  $W_R$  is displayed. The main effects plot reveals a significant linear relationship between the load and tempering temperature and the wear rate. For instance, lower tempering temperatures lead to less wear while higher tempering temperatures lead to more wear. The load factor showed a similar tendency; for instance, the wear rate was low under lower loading conditions while higher loading conditions resulted in a higher wear rate.

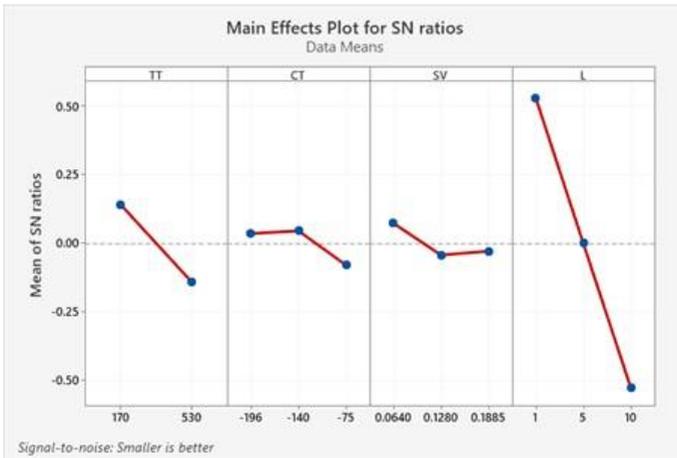


Figure 7. Main effects plot for SN ratios  $W_R$  [ $Al_2O_3$ ].

However, the relationship between the cryogenic treatment temperature and wear rate does not appear to be linear in this case; the wear rate was found to be higher for

specimens at -75 and -196 °C while being lower for steel treated at -140 °C. In the case of sliding velocity, higher sliding velocities cause an increase in wear rate, whereas lower sliding velocities cause a decrease in wear rate. The results of the ANOVA test indicate that load and tempering temperature are the two statistically significant factors that have the greatest impact on wear rate. As shown in **Table 5**, the most significant factor influencing wear rate is load (78.6%), followed by tempering temperature (8.45%).

Table 5. ANOVA table for WR [ $Al_2O_3$ ].

Source	DF	Seq SS	Adj SS	Adj MS	F	P	% C
TT	1	73.775	73.775	73.775	8.08	0.017	8.45
CT	2	11.862	11.862	5.931	0.65	0.543	1.36
SV	2	9.843	9.843	4.922	0.54	0.600	1.13
L	2	686.335	686.335	343.167	37.56	0.000	78.60
Residual Error	10	91.362	91.362	9.136			
Total	17	873.176					

The obtained results suggest that the wear rate is more influenced by the load and steel microstructure (or hardness). For instance, among all the specimens, samples treated at -140 °C and then tempered at 170 °C show the lowest wear rate and highest hardness. Therefore, one can observe an improvement in wear performance due to microstructure with higher population densities of small globular carbides, martensite refinement, and the precipitation of coherent transient carbides, particularly when combined with cryogenic treatment and tempering at 170 °C. These results are in excellent agreement with other researchers. Das et. Al, for instance, examined the wear resistance of D2 steel and found similar correlations with microstructure and wear rate [21], [22].

### 2.2.5 Bearing steel counterpart (100Cr6)

Commercially available bearing steel balls with 6 mm diameter were chosen as the second counterpart. The response variables considered are the friction coefficient (FC), wear rate ( $W_R$ ), and adhesion or counterpart material transfer (CMT) because it is anticipated that the medium hard counterpart will combine abrasive and adhesive wear. Calculations for wear volume and wear rate were made in accordance with ASTM G99-17 standard procedure, and a multi-parametric grey relation test which is described in Section 2.2 of the experimental procedure was used to optimize  $W_R$  and CMT together.

The experimental results for FC and corresponding SN ratios are shown in **Table 6**. The correlation between response variables in this case, FC, in response to various process settings is highlighted by the main effects plot, refer **Figure 8**. For instance, a direct linear relationship between tempering temperature and FC means that the FC value increases as tempering temperature does. On the other hand, there is no linear relationship between FC and cryogenic treatment.

Table 6. Experimental results for FC and SN ratios [100Cr6].

Exp.no	CT [°C]	TT [°C]	SV [m/s]	L [N]	FC	SN ratio
1	-75	170	0.064	1	0.666	3.53052
2	-75	170	0.128	5	0.597	4.48051
3	-75	170	0.1885	10	0.707	3.01161
4	-140	170	0.064	1	0.702	3.07326
5	-140	170	0.128	5	0.623	4.11024
6	-140	170	0.1885	10	0.715	2.91388
7	-196	170	0.064	5	0.565	4.95903
8	-196	170	0.128	10	0.619	4.16619
9	-196	170	0.1885	1	0.726	2.78127
10	-75	530	0.064	10	0.571	4.86728
11	-75	530	0.128	1	0.748	2.52197
12	-75	530	0.1885	5	0.737	2.65065
13	-140	530	0.064	5	0.608	4.32193
14	-140	530	0.128	10	0.652	3.71505
15	-140	530	0.1885	1	0.842	1.49376
16	-196	530	0.064	10	0.601	4.42251
17	-196	530	0.128	1	0.783	2.12476
18	-196	530	0.1885	5	0.717	2.88962

Furthermore, there is a strong positive correlation between sliding velocity and the FC, indicating that a rise in sliding velocity will result into an increase in the FC value. When the load is increased from 1 to 5 N, the FC value decreases, and when the load is increased to 10 N, the magnitude of the FC also increases, suggesting that load and the FC appear to have a strong negative linear relationship in the first scenario. It is clear from the main effects plot that these variations in FC values can be attributed to the final properties of the material and the tribological testing conditions.

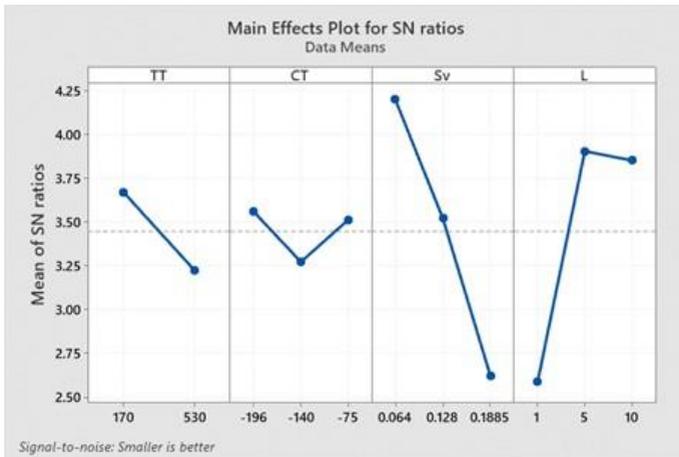


Figure 8. Main effects plot for SN ratios of FC [100Cr6].

The ANOVA results show that the most important factor influencing FC is sliding velocity, load, and tempering temperature. Sliding velocity contributes the most to FC (45.12%), followed by load (40.16%), and tempering temperature (5.42) as shown in **Table 7**. Load, sliding velocity, and tempering temperature are statistically significant factors, and cryogenic treatment was found to have no statistical significance. These findings are similar to those of the alumina counterpart for friction coefficient. The  $R^2$  value for ANOVA model is 92.41 % and the  $R^2$  (adj) value is 87.10 %. In other words, this model explains 92.41% of the total variation in the results of the experiments. The narrow difference and higher  $R^2$  and adj  $R^2$  values imply that the model proposed is highly accurate.

Table 7. ANOVA table for FC [100Cr6].

Source	DF	Seq SS	Adj SS	Adj MS	F	P	% C
TT	1	0.897	0.897	0.897	7.15	0.023	5.42
CT	2	0.282	0.282	0.141	1.12	0.363	1.71
SV	2	7.465	7.465	3.732	29.72	0.000	45.12
L	2	6.645	6.645	3.323	26.46	0.000	40.16
Residual Error	10	1.256	1.256	0.126			
Total	17	16.545					

The experimental results for  $W_R$  and CMT and their corresponding grey relation grades are shown in **Table 8**. Higher the grey relation grade better the performance of the material, hence, higher-the-better quality characteristic was used in Taguchi analysis for grey relation grade.

Table 8. Experimental results for Grey Relation Grade ( $W_R$  and CMT) [100Cr6].

Exp.no	CT [°C]	TT [°C]	SV [m/s]	L [N]	WR	CMT	GRG
1	-75	170	0.064	1	11.2	7.0	0.891
2	-75	170	0.128	5	20.5	17.0	0.572
3	-75	170	0.1885	10	31.1	13.9	0.554
4	-140	170	0.064	1	10.1	7.1	0.900
5	-140	170	0.128	5	26.4	19.2	0.509
6	-140	170	0.1885	10	31.0	19.1	0.486
7	-196	170	0.064	5	17.6	27.7	0.508
8	-196	170	0.128	10	23.3	29.4	0.455
9	-196	170	0.1885	1	4.7	31.4	0.667
10	-75	530	0.064	10	39.6	23.3	0.414
11	-75	530	0.128	1	7.7	19.9	0.686
12	-75	530	0.1885	5	24.6	22.1	0.493
13	-140	530	0.064	5	32.0	13.3	0.560
14	-140	530	0.128	10	33.2	21.0	0.458
15	-140	530	0.1885	1	10.8	13.9	1.49376
16	-196	530	0.064	10	51.4	18.9	4.42251
17	-196	530	0.128	1	9.0	13.9	2.12476
18	-196	530	0.1885	5	20.9	24.7	2.88962

The main effects plot for the grey relation grade is shown in **Figure 9**. It has been observed that the grey relation grade decreases with increase in tempering temperature, in other words, high tempering temperature deteriorates the wear performance of the steel. Additionally, the wear performance of materials treated at various cryogenic temperatures has varied. For example, among all the cryogenic temperatures, the wear performance of materials treated at -140°C in conjunction with low tempering treatment is the best. Also, it has been found that lower sliding velocities and loads tend to better wear performance and vice versa.

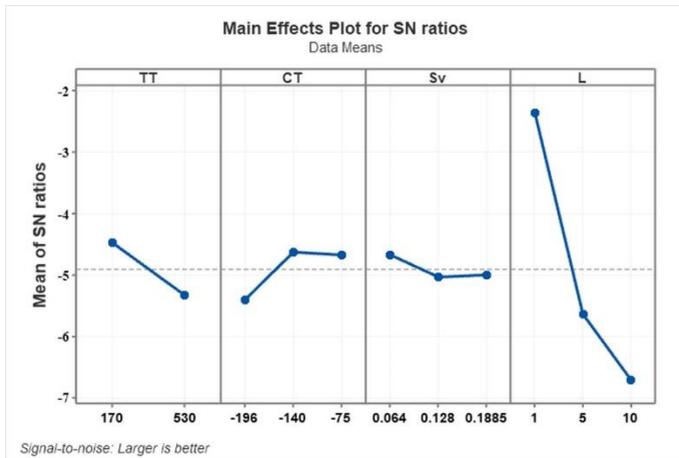


Figure 9. Main effects plot for the grey relation grade [100Cr6].

The ANOVA results, refer **Table 9** show that the load factor is the most significant parameter influencing the GRG with a contribution of 83%, followed by tempering temperature with 4 % contribution. The  $R^2$  value for the ANOVA model is 91.54 %, and the  $R^2$  (adj) value is 85.62 %, demonstrating that the model is highly accurate and can explain 91.54 % of the variance in GRG.

Table 9. ANOVA table Grey relation grade (GRG) [100Cr6].

Source	DF	Seq SS	Adj SS	Adj MS	F	P	% C
TT	1	3.284	3.284	3.284	5.28	0.04	4
CT	2	2.257	2.257	1.128	1.82	0.21	3
SV	2	0.480	0.480	0.240	0.39	0.69	1
L	2	61.215	61.215	30.608	49.26	0.00	83
Residual Error	10	6.213	6.213	0.621			
Total	17	73.45					

The contour plot of grey relation grade as a function of load and material hardness is shown in **Figure 10**. There is a strong correlation between the grey relation grade, applied load, and material hardness. With increasing load, the magnitude of the grey relation grade

decreases. This implies that an increase in load causes an increase in both  $W_R$  and CMT regardless of the cryogenic treatment used. Furthermore, as the material's hardness increases, the  $W_R$  and CMT decrease. The combination of cryogenic treatment at  $-140\text{ }^\circ\text{C}$ , tempering temperature at  $170\text{ }^\circ\text{C}$ , in combination with low sliding velocities and load results in the highest GRG (lowest  $W_R$  and CMT), refer **Table 8**. The obtained results indicate that the GRG is affected by the load and steel microstructure, as evidenced by hardness. The highest GRG obtained with this combination of heat treatment parameters is logical because the investigated steel had the highest hardness values after cryogenic treatment at  $-140\text{ }^\circ\text{C}$  followed by tempering at  $170\text{ }^\circ\text{C}$ . Furthermore, this heat treatment schedule maximised the number and population density of SGCs, as shown in **Figure 3b**, which is a critical factor influencing the GRG.

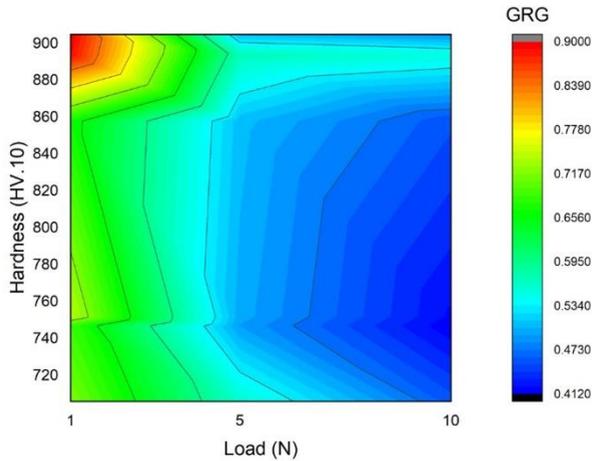


Figure 10. Contour plot for GRG vs hardness and load [100Cr6].

It is also worth noting that the results obtained are consistent with those of other researchers. Akhbarizadeh et al. [23] investigated the wear behaviour of cryo-treated AISI D3 steel against comparable counterpart materials and discovered that the increased amount of SGCs in the microstructure improved wear resistance and hardness. Das et al. [21], [22], [24] compared the wear performance of cryotreated AISI D2 steel to that of tungsten carbide counterpart. They came to very similar conclusions, that the wear performance of cryotreated steel improves as the number of SGCs increases. The best wear performance was obtained at the peak of SGCs count, which was established after 36 hours of treatment at boiling nitrogen ( $-196\text{ }^\circ\text{C}$ ). The number and density of SGCs also play an important role in explaining differences in counterpart material transfer. Cryogenic treatment at  $-140\text{ }^\circ\text{C}$  results in the highest population density of SGCs carbides, as shown in **Figure 3b**. Besides that, Fontalvo et al. [25] investigated the impact of carbides content and type on the adhesive wear of powder metallurgy (PM) high speed steel (AISI M4) and found that the

two main factors influencing the adhesive wear behaviour are the carbides content and the distance between carbide particles (which is inversely proportional to their population density). When Gaard et al. [26] investigated the impact of microstructure on the initiation of galling and wear mechanisms of AISI D2 steel under dry sliding configuration against a carbon sheet, they discovered a similar effect. Therefore, it makes sense that applying a cryogenic treatment at a temperature of -140 °C followed by low-temperature tempering will result in the greatest improvement in the overall wear performance of the Cr-V ledeburitic tool steel.

### 2.2.6 Bronze counterpart (CuSn6)

In order to simulate a pure adhesive wear, 6 mm diameter CuSn6 balls are used as a counterpart. Because the CuSn6 has a hardness of 74 HV 10, which is significantly lower than the experimental tool steel's hardness, adhesion is expected to be significant. Therefore, the wear performance of this tribosystem cannot be assessed using ASTM G99-17. As a viable alternative, the amount of counterpart material transfer (CMT%) or (adhesion%) was chosen for investigations. To evaluate the degree of adhesion, it was calculated what percentage of the worn track was covered by the counterpart material. Twenty randomly chosen SEM images of the worn surfaces were used in the adhesion calculations. Friction coefficient and adhesion are taken into account as the response variables in this situation.

**Table 10** exhibits the friction coefficient and adhesion experimental results along with the corresponding SN ratios, and **Figures 11** and **12** display the main effects plot for the corresponding SN ratios.

*Table 10. Experimental results for FC, Adhesion (CMT%), and their SN ratios [CuSn6].*

S. No	CT [°C]	TT [°C]	SV [m/s]	L [N]	FC	SN ratio	CMT%	SN ratio
1	-75	170	0.064	1	1.107	-0.88	37.0	-31.358
2	-75	170	0.128	5	0.904	0.87	90.9	-39.172
3	-75	170	0.1885	10	0.834	1.57	93.2	-39.388
4	-140	170	0.064	1	0.932	0.61	61.0	-35.708
5	-140	170	0.128	5	0.889	1.02	90.3	-39.110
6	-140	170	0.1885	10	0.875	1.15	97.3	-39.764
7	-196	170	0.064	5	0.932	0.61	71.7	-37.104
8	-196	170	0.128	10	0.880	1.11	95.2	-39.576
9	-196	170	0.1885	1	1.016	-0.13	18.6	-25.404
10	-75	530	0.064	10	0.941	0.52	60.3	-35.607
11	-75	530	0.128	1	1.015	-0.12	27.2	-28.682
12	-75	530	0.1885	5	1.015	-0.12	51.0	-34.147
13	-140	530	0.064	5	0.985	0.13	27.4	-28.756
14	-140	530	0.128	10	0.939	0.54	88.0	-38.890
15	-140	530	0.1885	1	0.974	0.22	23.8	-27.542
16	-196	530	0.064	10	0.938	0.55	61.4	-35.761
17	-196	530	0.128	1	0.935	0.58	12.5	-21.962
18	-196	530	0.1885	5	0.959	0.36	53.3	-34.526

According to the main effects graph, the tempering temperature and load have a significant impact on the FC value. For instance, a higher load and lower tempering temperature lower the FC value, and vice versa. However, it doesn't seem as though there is a linear relationship between the FC values and sliding velocity or cryogenic treatment. However, lower friction values are favoured when materials are subjected to cryogenic treatment at -140°C and a medium sliding speed of 0.128 m/s.

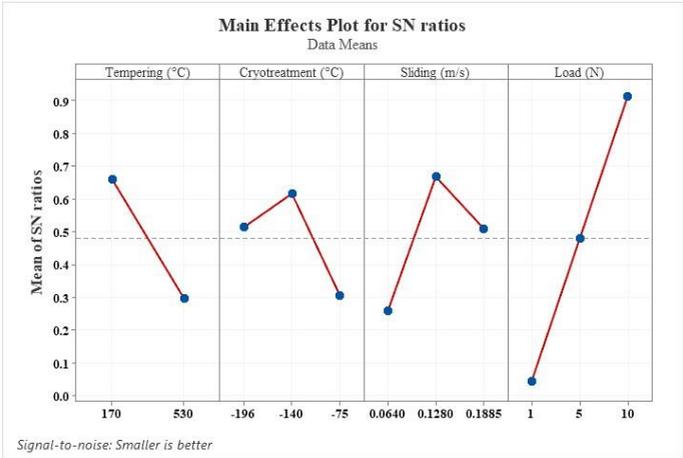


Figure 11. Main effects plot for SN ratios of FC [CuSn6].

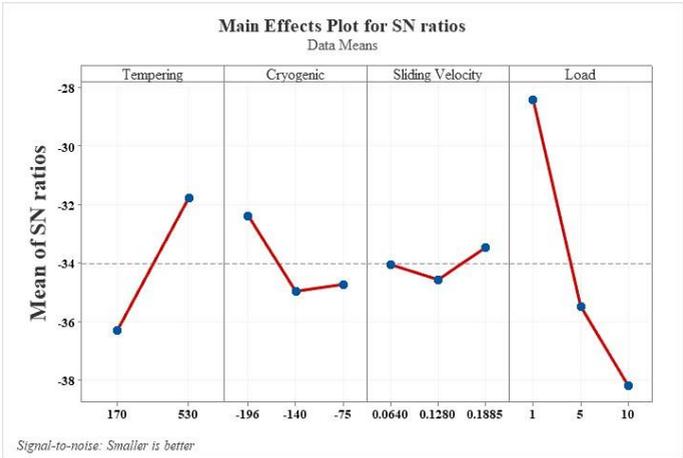


Figure 12. Main effects plot for SN ratios of adhesion [CuSn6].

The adhesion appears to be dependent on tempering temperature and load, and the main effects suggest that these two variables have significant linear relationships. For

instance, lower adhesion is produced by higher tempering temperatures, and vice versa. Adhesion also rises with increasing load. The adhesion is also moderately affected by cryogenic treatment; for instance, adhesion is lower at -196°C than at other temperatures. On the other hand, the adhesion appears to be little affected by sliding velocity. Additionally, ANOVA studies were conducted to confirm the statistical significance, and the results are shown in **Tables 11** and **12** for friction coefficient and adhesion, respectively.

*Table 11. ANOVA table for friction coefficient [CuSn6].*

Source	DF	Seq SS	Adj SS	Adj MS	F	P	% C
TT	1	0.5934	0.5934	0.5394	2.84	0.123	10.3
CT	2	0.2996	0.2996	0.1498	0.72	0.512	5.2
SV	2	0.5106	0.5106	0.2553	1.22	0.335	8.9
L	2	2.2564	2.2564	1.1282	5.39	0.026	39.2
Residual Error	10	2.0914	2.0914	0.2091			
Total	17	5.7514					

*Table 12. ANOVA table for adhesion [CuSn6].*

Source	DF	Seq SS	Adj SS	Adj MS	F	P	% C
TT	1	92.071	92.071	92.071	10.27	0.009	18.0
CT	2	24.271	24.271	12.136	1.35	0.302	4.7
SV	2	3.657	3.657	1.828	0.2	0.819	0.7
L	2	302.303	302.303	151.152	16.87	0.001	59.1
Residual Error	10	89.621	89.621	8.962			
Total	17	511.924					

The only statistically significant factor for friction coefficient, according to ANOVA tests, is the load, which contributes 39.2% of the total variance. The remaining factors are not statistically significant. The statistically significant variables in the case of adhesion are tempering temperature, which contributes 18%, and load, with a contribution of 59.1%.

In conclusion, the contact stresses may be the major factor in why the friction coefficient is more load dependent than material dependent. As the load rises, the contact stresses rise along with them, increasing the counterpart's plastic deformation as a result. This increases adhesion on the wear track and lowers the friction coefficient due to the lubrication effect. Adhesion, on the other hand, is more influenced by the applied treatments and load. When compared to other treatments, it was discovered that cryogenic treatment at -196 °C in conjunction with high tempering at 530 °C had the minimum adhesion. There are three major factors that could influence the variations in adhesion levels. As was already mentioned, the first one is the load. The retained austenite amount is the second factor, lower adhesion might be explained by a drop in retained austenite amounts during cryogenic treatment. The third factor to take into account is the impact of carbide population densities on adhesion (or galling resistance). Recent research has shown that, when compared to conventional heat treatment, short-term cryogenic treatments followed by high-temperature tempering increase the Cr-V ledeburitic tool steel anti-galling resistance [27]. The galling

resistance of tool steels may benefit from an increased number of carbides, according to Gaard et al. [28] and Fontalvo et al. [25]. However, Fontalvo et al. [25] also stated that there is an ideal carbide amount at which tool steel galling resistance is maximised; lower and higher carbide amounts both result in worse galling resistance.

Thus, it can be inferred that the SGCs quantity obtained by the cryogenic treatment at  $-196\text{ }^{\circ}\text{C}$  is almost an "optimum" value to obtain the best anti-galling properties of the examined steel, and that further increase of SGCs count (by applying treatment at either  $-75$  or  $-140\text{ }^{\circ}\text{C}$ ) may be detrimental to the galling resistance of Cr-V ledeburitic tool steel. The best anti-galling characteristics of  $-196\text{ }^{\circ}\text{C}$  cryotreated and high-temperature tempered Vanadis 6 steel may be explained by this factor, along with the lowest retained austenite amount.

### 2.2.7 Cryogenic treatment (CT) vs. conventional treatment (CHT)

The research shows that cryogenic treatment (CT) improves the microstructure, hardness, and wear resistance of Cr-V ledeburitic steel. According to the results of earlier research, when compared to 100Cr6 and  $\text{Al}_2\text{O}_3$  counterparts, cryogenic treatment at  $-140\text{ }^{\circ}\text{C}$  for 17 hours after tempering at  $170\text{ }^{\circ}\text{C}$  is the best temperature for achieving improved wear performance. As a result, using hard alumina counterpart, the abrasive wear characteristics of cryotreated specimens were compared to those of conventionally treated specimens. The load, sliding velocity, and sliding distance were the input variables used in the wear experiments, which were conducted using the Taguchi L9 orthogonal array design.

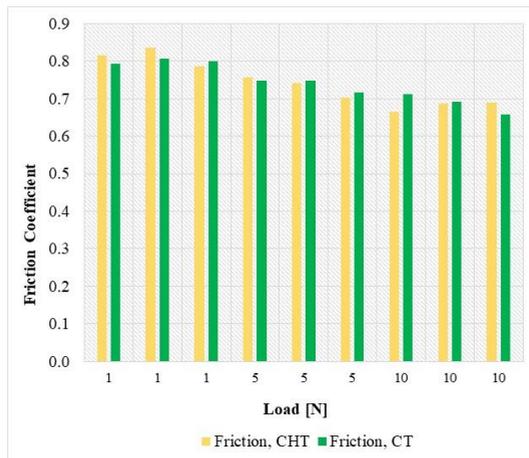


Figure 13. Friction coefficient values for CHT and CT specimens for all experiments.

Wear tests were conducted on mirror polished specimens prepared by standard metallographic procedures. **Figure 13** shows the mean friction coefficient values for CHT

and CT specimens at various wear loads, sliding speeds, and sliding distances. Two main tendencies can be derived from the friction graph. First, the main factor affecting friction coefficient is load. For instance, friction coefficient for both treatments, particularly for the CT specimens, decreases with increasing load. Second, no noticeable difference between the friction coefficients of the CHT and CT specimens was found. However, at lower and higher loads, the friction coefficient of CT specimens found less compared to conventionally treated specimens. For instance, in the first experiment the mean friction coefficient value for CT specimen is 0.793 at 1 N load, 0.064 sliding speed, at a sliding distance of 100 m. In the case CHT specimen the mean friction coefficient value found to be 0.815 for similar conditions. In this scenario it can be summarised that the treatment of the samples almost has no effect on coefficient friction and friction coefficient decreases with increase in loading. These findings are highly consistent with those of other researchers [29]. They demonstrated that increasing the load causes the friction coefficient to decrease and that cryogenic treatment has no impact on friction. In **Figure 14**, the wear rate ( $W_R$ ) of CHT and CT specimens is shown for all the experiments.

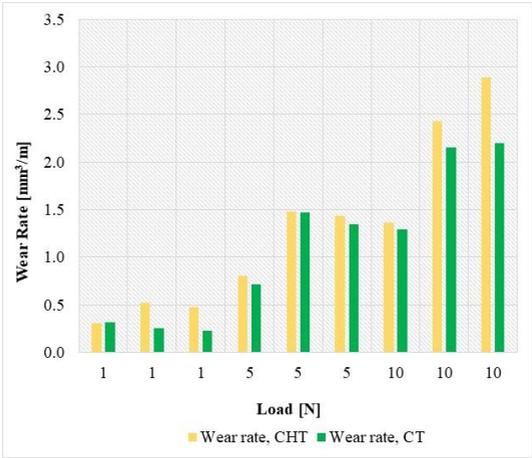


Figure 14. Wear rate of CHT and CT specimens for all experiments.

The wear graph showed that both conventional and cryotreated specimens had an increasing wear rate as the applied load increased. In every testing scenario, cryotreated specimens showed lower wear rates. When samples were worn under comparable testing conditions, such as at 1 N, 0.1885 m/s, and 200 m, the CHT sample showed the highest wear rate (0.479 mm<sup>3</sup>/Nm), while the CT specimen showed the lowest wear rate (0.226 mm<sup>3</sup>/Nm). The main reasons for this enhanced wear performance include increased population densities of small globular carbides, higher hardness values (933 HV10), and overall microstructure refinement. The other research and these findings have an excellent level of agreement. In testing the wear performance of various cold work tool steels, for

instance, Podgornik et al. [29] and Pellizzari et al. [30] arrived at the conclusion that the improved wear performance of cryotreated specimens is due to the microstructural changes produced at cryogenic temperatures.

### 2.2.8 Wear mechanisms

All the worn surfaces were subjected to thorough SEM analysis to determine the wear mechanism. For any specimens that have been cryotreated, the wear mechanism for the hard alumina ( $Al_2O_3$ ) counterpart is abrasive, as shown in **Figure 15**. However, compared to other treatments at higher load and sliding velocities, the damage to the worn surface, such as the abrasive marks of cryotreated specimens at  $-140\text{ }^\circ\text{C}$ , is less severe. This is also evidenced by the lower wear rate of samples treated at  $-140\text{ }^\circ\text{C}$  in combination with low temperature tempering.

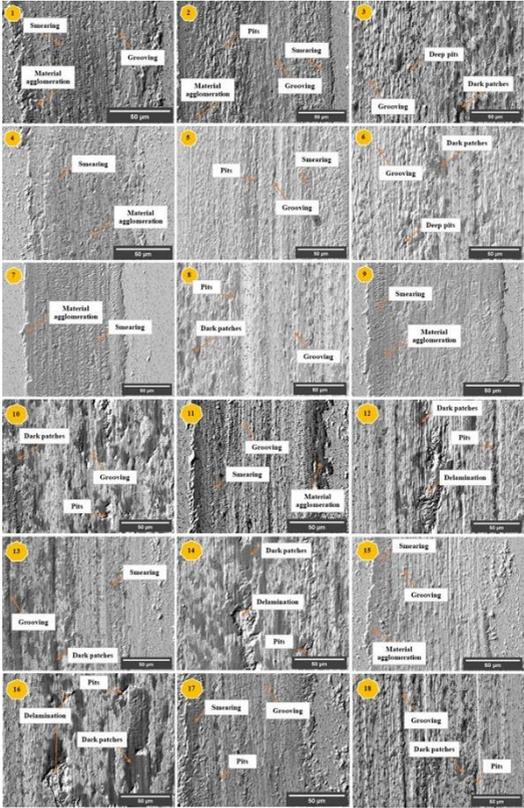
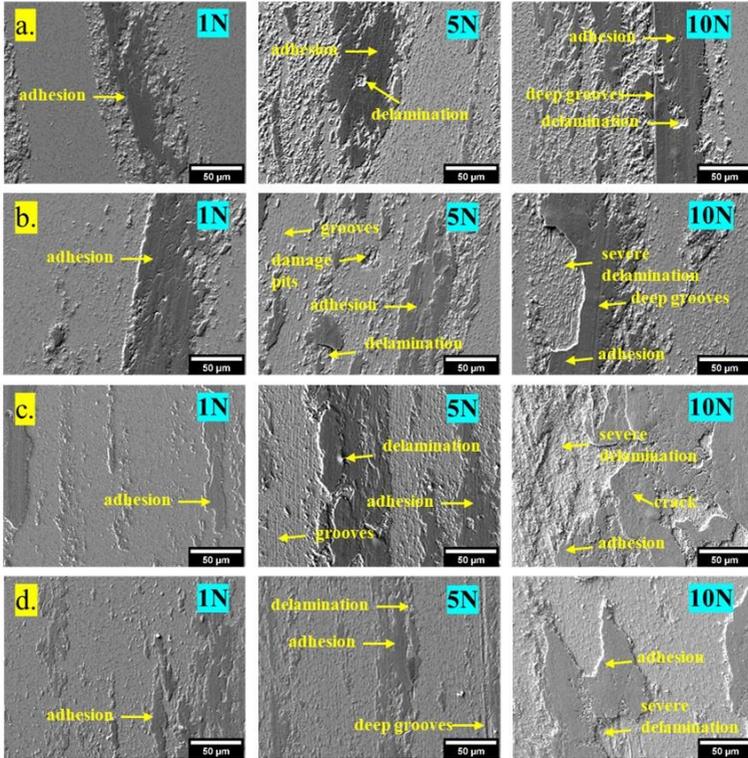


Figure 15. Abrasive wear mechanism of cryotreated specimens [ $Al_2O_3$ ].

As shown in **Figure 16**, adhesive wear and a combination of adhesive and abrasive wear are the main wear mechanisms in the case of bearing steel (100Cr6) counterpart. Higher loads and sliding speeds, however, cause the wear mechanism to switch to delamination, increasing wear rates.



*Figure 16. Adhesive wear mechanism of cryotreated samples against 100Cr6 steel showing delamination at higher sliding velocities.*

Despite the similarity of the wear mechanisms, cryogenic treatment at  $-140^{\circ}\text{C}$  in conjunction with low temperature tempering treatment produces a higher grey relation grade, which results in a lower wear rate and adhesion or counterpart material transfer. The higher hardness and population densities of small globular carbides, combined with cryogenic treatment at  $-140^{\circ}\text{C}$  and tempering treatment at  $170^{\circ}\text{C}$ , lead to better wear performance for abrasive and mixed wear mechanisms. In the case of CuSn6 counterpart, the wear mechanism is adhesive, and the cryogenic treatment at  $-196^{\circ}\text{C}$  and tempering treatment at  $530^{\circ}\text{C}$  result in better adhesion or galling resistance. The SEM micrographs of exposed surfaces showing difference in adhesion in reference to tempering temperature at different loads is shown in **Figure 17**.

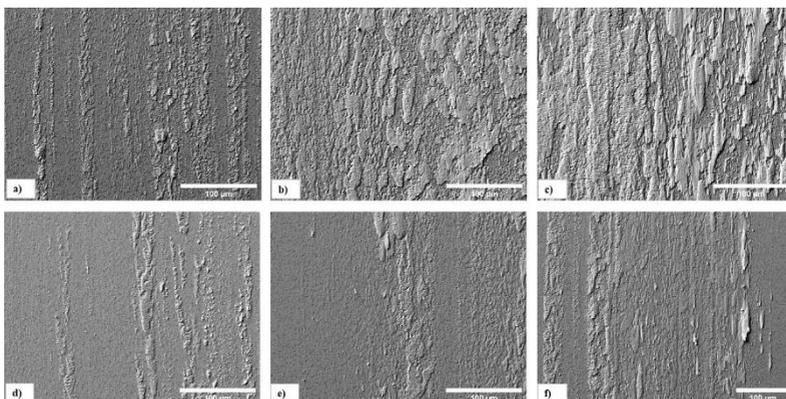


Figure 17. Exposed surface SEM images of (-75) low and high temperature tempered specimens at different loading conditions: (a) 1 N, (b) 5 N, and (c) 10 N, tempered at 530 °C (d) 1 N, (e) 5 N, and (f) 10 N. [CuSn6].

In **Figure 18**, the SEM images of worn surfaces of conventional and cryogenically treated specimens at 10N load when tested against alumina ( $\text{Al}_2\text{O}_3$ ) counterpart is shown. The SEM images show that the wear mechanism is the same for both treatments, for example, the abrasive wear mechanism with delamination, but the severity of abrasion and delamination was less severe in samples that were cryotreated at  $-140^\circ\text{C}$  than in specimens that were treated conventionally. The population densities of small globular carbides and matrix hardness are the main contributors to this contribution, as the latter reduces delamination and enhances wear performance.

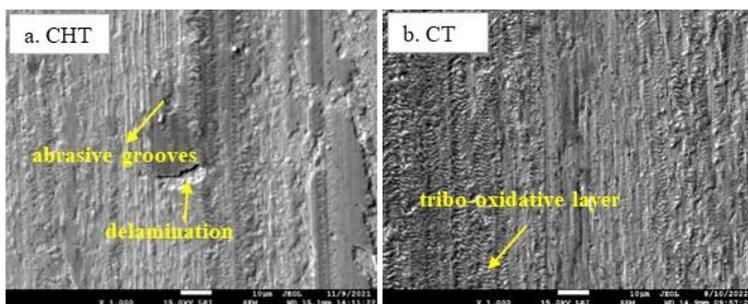


Figure 18. Wear mechanism of CHT and CT specimens [ $\text{Al}_2\text{O}_3$ ].

In conclusion, it has been found that when using various counterparts, different cryogenic and tempering treatments cause different wear behaviours of Cr-V ledeburitic tool steel. The wear performance is better with CT specimens at  $-140^\circ\text{C}$  combined with low temperature tempering at  $170^\circ\text{C}$  than with other treatments for abrasive wear mechanisms and for mixed abrasive and adhesive wear mechanisms. Additionally, in this instance, cryogenic treatment is better than conventional treatment. In the case of adhesive wear,

cryogenic treatment at  $-196^{\circ}\text{C}$  and in combination with high temperature tempering at  $530^{\circ}\text{C}$  improves the adhesion or galling resistance.

## CONCLUSIONS

The microstructure, hardness, and tribological performance of Vanadis 6 steel were investigated against counterparts made of alumina, ball bearing steel (100Cr6), and CuSn6 bronze. Various cryogenic treatments, tempering regimes, and wear testing conditions were investigated. The investigations led to the following findings:

- The microstructure of Vanadis 6 steel contains martensitic matrix with uniform distribution of vanadium rich eutectic carbides (ECs), chromium rich secondary carbides (SCs), and small globular carbides of cementite type.
- The specimens that were tempered at 170 °C had a higher hardness than those that were tempered at 530 °C. Cryogenic treatment at -140 °C produced the highest hardness.
- The friction coefficient is more dependent on the load and sliding velocity than the treatment of the material.
- The load, tempering temperature, and sliding velocity have a greater impact on the wear rate. Sliding velocity, however, is not statistically significant.
- The steel after cryogenic treatment at -140 °C combined with tempering treatment at 170 °C has the best tribological performance when tested with alumina counterpart.
- The wear mechanism is abrasive when tested against alumina counterpart, and samples that were treated at -140°C showed less severe abrasion.
- When tested against bearing steel counterpart, cryogenic treatment at -140°C in combination with tempering treatment at 170°C produces the best tribological performance.
- The wear mechanism is a mixed adhesive and abrasive when tested against bearing steel (100Cr6) counterpart exhibiting delamination at higher loads and sliding speeds.
- When tested against CuSn6 counterpart, cryogenic treatment at -196°C in combination with tempering treatment at 530°C provides the examined steel the best adhesion or galling resistance.
- The wear mechanism is complete adhesion with no sign of abrasion when tested against soft CuSn6 counterpart.
- The abrasive wear performance of the Cr-V ledeburitic tool steel when tested against alumina counterpart is superior to conventional treatment when treated at -140°C in combination with tempering at 170°C.

The findings in this thesis may help to establish the foundational knowledge for cold forming applications in the tooling sector. According to the application of tool steel in a variety of wear mechanisms, including abrasive, adhesive, and mixed wear of abrasive and adhesive, the results related to tribological performance obtained in this thesis may simplify the selection of heat treatment parameters.

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1. Yarasu, V., Jurci, P., Hornik, J. (2022). Dry sliding tribological behaviour of cryotreated Cr-V ledeburitic tool steel against CuSn6 counterpart. *Steel Research International*. <https://doi.org/10.1002/srin.202200453>. [Category: ADC (A)].
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6. Yarasu, V., Rausa, L., Jurči, P., Horník, J. (2022). The effect of cryogenic treatment on wear performance of Vanadis 6 steel. In *International Doctoral Seminar 2022*. Trnava: AlumniPress, p. 338--346. ISBN 978-80-8096-292-0. [Category: AFC (C)].
7. Yarasu, V., Jurci, P., Hornik, J. (2022). A comparative study on wear behaviour of conventionally and cryogenically treated cold work tool steel. In *AMPT2022 Conference Proceedings*; Portorož, Slovenia. [Category: AFC (C)].

## ABSTRACT

YARASU, Venu: *Study of changes in wear resistance of Cr-V ledeburitic tool steel due to cryogenic processing*. [Dissertation thesis]- Slovak University of Technology Bratislava. Faculty of Materials Science and Technology; Institute of Materials Science- Supervisor: Prof. Ing. Peter Jurci, PhD - Trnava: MTF STU, 2022.

This thesis deals with the tribological behaviour including the wear mechanism of Cr-V ledeburitic tool steel Vanadis 6 due to cryogenic processing. It is the fact that the cryogenic treatment provides some additional benefits with respect to the microstructure and properties of ledeburitic tool steels over conventional heat treatment is commonly known. However, the choice of optimal regime of this kind is still under debate. Nowadays, the temperature of boiling nitrogen ( $-196\text{ }^{\circ}\text{C}$ ), and the duration ranging between 17 and 36 h is the most widely accepted regime of cryogenic treatment. However, some authors recommend higher treatment temperature (i.e., around  $-140\text{ }^{\circ}\text{C}$ ) while others suggested rather lower temperatures of this processing, i.e., the boiling temperature of liquid helium ( $-269\text{ }^{\circ}\text{C}$ ), for instance. Unfortunately, there is not any relevant study on the effect of the cryogenic treatment at such a very low temperatures on the wear resistance of the materials. The thesis will be focused to the investigation of changes in the wear resistance of Cr-V ledeburitic cold work tool steel Vanadis 6 when subjected to the cryogenic treatment at different temperatures followed by tempering treatment at 170 and 530  $^{\circ}\text{C}$ . The tribological behaviour of Cr-V ledeburitic cold work tool steel in pin-on-disk configuration is studied using different counterpart's material (soft, medium, and very hard material). The show that when the specimens are treated at  $-140\text{ }^{\circ}\text{C}$  in combination with low temperature tempering (170  $^{\circ}\text{C}$ ), Vanadis 6 steel has superior wear performance in sliding against hard alumina and medium hard bearing steel counterparts. The predominant wear mechanism is abrasive with an alumina counterpart, and a mixed wear mechanism of abrasive and adhesive is found dominant with a bearing steel counterpart. When a soft counterpart is employed, specimens treated at  $-196\text{ }^{\circ}\text{C}$  in conjunction with high temperature tempering (530  $^{\circ}\text{C}$ ) exhibit the highest galling resistance and the wear mechanism is either adhesion or galling. The results that were seen are consistent with the cryogenic treatment's effects on the microstructure, which include a decrease in retained austenite and an increase in the population densities of small globular carbides. Although load- and sliding-speed-dependent trends were noticed, cryogenic treatments had no impact on friction coefficient.

### **Keywords:**

Cr-V ledeburitic tool steel, Conventional treatment, Cryogenic treatment, Wear behaviour, Taguchi technique, Wear mechanisms.

## SÚHRN

YARASU, Venu: *Štúdium zmien odolnosti Cr-V ledeburitickej nástrojovej ocele proti opotrebeniu v dôsledku kryogénneho spracovania*. [Dizertačná práca]- Slovenská technická univerzita v Bratislave. Materiálovo technologická fakulta so sídlom v Trnave; Ústav materiálov- Vedúci práce: Prof. Ing. Peter Jurci, PhD - Trnava: MTF STU, 2022.

Táto práca sa zaoberá tribologickým správaním vrátane mechanizmu opotrebenia Cr-V ledeburitickej nástrojovej ocele Vanadis 6 v dôsledku kryogénneho spracovania. Je všeobecne známe, že kryogénne spracovanie vedie k určitým výhodným zmenám v mikroštruktúre a vlastnostiach ledeburitických nástrojových ocelí v porovnaní s konvenčným tepelným spracovaním. Výber optimálneho režimu tohto druhu spracovania je však stále predmetom diskusií. V súčasnosti je najrozšírenejším režimom kryogénneho spracovania teplota kvapalného dusíka (-196 °C) a trvanie v rozmedzí 17 až 36 hodín. Niektorí autori však odporúčajú vyššiu teplotu spracovania (t.j. okolo -140 °C), iní navrhujú skôr nižšie teploty tohto spracovania, teda napríklad teplotu varu tekutého hélia (-269 °C). Bohužiaľ neexistuje žiadna relevantná štúdia o vplyve kryogénneho spracovania pri takých veľmi nízkych teplotách na odolnosť materiálov proti opotrebovaniu. Dizertačná práca bude zameraná na skúmanie zmien odolnosti proti opotrebeniu Cr-V ledeburitickej nástrojovej ocele Vanadis 6 na tvárnenie za studena v dôsledku kryogénneho spracovania pri rôznych teplotách s následným popúšťaním pri 170 alebo 530 °C. Tribologické správanie Cr-V ledeburitickej nástrojovej ocele na prácu za studena v konfigurácii pin-on-disk je v práci študované s použitím rôznych materiálov protikusov (mäkký, stredne tvrdý a veľmi tvrdý materiál). Výsledky ukazujú, že keď sú vzorky ošetrené pri -140 °C v kombinácii s nízkoteplotným popúšťaním (170 °C), oceľ Vanadis 6 má vynikajúcu odolnosť proti opotrebovaniu pri suchom trení proti tvrdému oxidu hlinitému a stredne tvrdé ložiskové oceli. V prípade protikusu z oxidu hlinitého je prevládajúci mechanizmus opotrebovania abrazívny, a u protikusu z ocele 100Cr6 prevláda mechanizmus zmiešaného abrazívno/adhezívneho opotrebovania. Pri použití mäkkého protikusu z bronzu, vykazujú najlepšie tribologické vlastnosti vzorky spracované pri -196 °C v spojení s vysokoteplotným popúšťaním (530 °C). Hlavným mechanizmom opotrebovania je adhézia spojená s nalepovaním protikusu na nástrojovú ocel. Dosiagnuté výsledky sú v súlade s účinkami kryogénneho spracovania na mikroštruktúru, ktoré zahŕňajú zníženie obsahu zvyškového austenitu a zvýšenie množstva malých globulárnych karbidov. Hoci boli zaznamenané trendy závislé od zaťaženia a rýchlosti posuvu, kryogénne úpravy nemali žiadny vplyv na koeficient trenia.

### **Kľúčové slová:**

Cr-V ledeburitická nástrojová oceľ, konvenčné spracovanie, kryogénne spracovanie, tribologické správanie, technika Taguchi, mechanizmy opotrebovania.