

Iterative Taguchi Analysis: Optimizing the Austenite Content and Hardness In 52100 Steel

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Three iterations of Taguchi designed experiments and analyses were used to determine optimal thermal treatments for minimizing retained austenite content while maximizing Rockwell hardness (HRC) in AISI 52100 bearing steel. Experimental variables chosen for this study included austenitizing and tempering temperatures, tempering time and cold treatment. After one iteration, tempering temperature and cold treatment were seen to have the greatest effect on austenite content while austenitizing and tempering temperatures had the greatest influence on hardness. After the second and third experimental iterations, two thermal treatments were noted each producing hardness of 58-59 HRC in combination with zero retained austenite as measured by x-ray diffraction.

Keywords 52100 steel, austenite, design of experiment, heat treating, Taguchi

1. Introduction

Taguchi[1,2] design of experiment (DOE) methods incorporate fractional factorial matrixes or orthogonal arrays to minimize the number of experiments required to achieve a given set of performance characteristics. Iterative Taguchi experiments can be designed to systematically approach optimal parameters for a complicated process or as a quality assurance tool to identify the important parameters to monitor for statistical process control. The Taguchi experimental approach allows a statistically sound experiment to be completed, while investigating a minimum number of possible combinations of parameters or factors. A Taguchi experiment can be accomplished in a timely manner and at a reduced cost with results comparable to a full factorial experiment.

Determination of appropriate times and temperatures for a heat-treating procedure that will achieve both low retained austenite and a high hardness can appear initially to require extensive, if not prohibitive, experimentation. Fortunately, Taguchi analysis provides an efficient and effective means of achieving these goals. If retained austenite transforms during service, the associated nominal 4 vol.% increase produces distortion, which can lead to seizure and premature failure. The austenite content is commonly limited to less than 3% for critical precision bearings and 15% for some gearing applications. Higher hardness is generally associated with improved fatigue strength and resistance to spalling failure and wear. To minimize retained austenite and maximize hardness simultaneously, appropriate austenitizing, quenching, and perhaps cryogenic cooling procedures must be determined.

This paper describes an application of a Taguchi analysis to reach an optimal set of processing parameters through a simple and inexpensive iterative process that could be used to develop heat-treatment processing parameters for a wide variety of alloys. The heat treatment of critical bearing components fabricated from 52100 steel requiring both minimal austenite content and high hardness for dimensional stability in service, wear resistance, and load bearing strength was chosen to demonstrate the approach.

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2. Experimental Design and Technique

The objective of the following study was to determine how an iterative Taguchi experimental design could be used to systematically optimize a complicated heat-treatment process that has several potential variables. The maximum amount of retained austenite, the face-centered cubic (fcc) form of iron and carbon commonly found in hardened steel, can be required to be as low as 3% for some bearing components because of its effect on the dimensional stability when in service. A high hardness or the resistance to penetration is also important because of its association with wear resistance and load bearing strength. Therefore, a DOE was assembled for the heat treatment with the goal of simultaneously yielding the highest hardness and the lowest level of retained austenite. A widely used bearing alloy, 52100 steel, was selected to demonstrate the method, although a wide range of iron-base alloys could have been selected.

The four parameters or factors identified as primarily affecting the retained austenite and/or hardness were the austenitizing temperature, tempering temperature, tempering time, and cryogenic or cold treatment.[3,4] These factors are normally specified in heat-treating references as being the most important. The austenitizing temperature is the temperature to which steel is heated in order to transform the body-centered cubic ferrite to homogeneous fcc austenite, increasing the stability of carbon. Austenitizing is performed prior to the quenching operation that hardens the steel, trapping the carbon to form martensite. The temperature specified for austenitizing is the maximum temperature to which the material is heated during the heat-treating process. The tempering operation, performed for a predetermined time and temperature below the martensitic transformation temperature, normally has the effect of reducing the

Table 1 Factor and level descriptions for Taguchi DOE A

	Factors	Level 1	Level 2
A	Austenitizing temperature	774 °C (1425 °F)	871 °C (1600 °F)
B	Tempering temperature	93 °C (200 °F)	343 °C (650 °F)
D	Tempering time	1 h	4 h
H	Cold treatment	None	1 h
	Interactions		
C	Austenitizing temperature vs tempering temperature	…	…
E	Austenitizing temperature vs tempering time	…	…
F	Tempering temperature vs tempering time	…	…
I	Austenitizing temperature vs cold treatment	…	…
J	Tempering temperature vs cold treatment	…	…
L	Tempering time vs cold treatment	…	…

hardness, increasing the ductility, and decreasing the amount of retained austenite. The cold treatment, performed during this investigation, in liquid nitrogen at a temperature of −210 °C, is a method sometimes used to reduce the amount of retained austenite.

To initially identify any interactions that may take place among the factors, an L16 $(2)^{15}$ array, with two levels for each factor, was chosen for the initial DOE (DOE A). The L16 $(2)^{15}$ designation refers to the number of experiments (16), the number of levels for each factor (2), and the number of factors or interactions (15). A full factorial experiment would consist of $(2)^{15}$, or 32,768 experiments, as compared to the Taguchi experiment requiring only 16 experiments. All interactions are considered for the initial screening DOE to eliminate any confounding of the matrix columns that make interpretation of the results difficult. An interaction is defined as an occurrence where the total effect is greater than the sum of the total effects taken independently. The recommended heat treatment[5] commonly performed for 52100 steel was the basis for selection of the initial two levels for each factor. The two levels should represent reasonable extremes for each of the selected factors, especially for the initial DOE.

Once the possible interactions were identified, an L9 $(3)^4$ array, employing nine experiments, three levels for each of the remaining four factors or interactions, was chosen for a second DOE (DOE B) to increase the number of levels for each factor and to decrease the number of experiments. Finally, a third Taguchi experiment (DOE C) was performed to refine the results of the second experiment and to approach the optimal heat-treating parameters. During the third experiment, the best values from the second Taguchi experiment were used as nominal levels to set each factor. The ranges between the high and low levels were also decreased for DOE C.

The 52100 steel bar stock used during this investigation was purchased in an annealed condition with an initial hardness less than 25 HRC and no measurable retained austenite. Disks that were approximately 0.5 in. thick were sectioned from the bar stock to be used in the analysis. A total of 16 disks were used for the first experiment, and a total of 9 disks were used for each of the second and third experiments. The hardness and retained austenite measurements were made on the flat face of each specimen after a mechanical polish to a 6 μm diamond finish.

Retained austenite measurements are determined by quantitative microscopic examination if the austenite is high, usually above about 15%. Since the austenite content can be very low in bearing steels, a more accurate x-ray diffraction technique was used during this investigation. The retained austenite measurements were made by x-ray diffraction in accordance with ASTM E975 and SAE SP-453, using the direct comparison method of Averbach and Cohen.[5] The unit cell volume and the chemical composition of 52100 steel were used to calculate the intensity factors, *R*.[6]

The integrated intensity of each austenite and ferrite/martensite peak was measured using chromium *K* radiation. The use of multiple diffraction peaks from each phase minimizes the possible effects of preferred orientation and coarse grain size. Four independent volume percent retained austenite values were calculated from the *R* ratios and the total integrated intensities of the austenite (200) and (220) and ferrite/martensite (200) and (211) diffraction peaks.

A Miller fixture[7] was used to minimize the influence of preferred orientation and grain size. The Miller fixture rotates the specimen around the surface normal and oscillates (±45°) perpendicular to the diffraction plane.

The Rockwell C hardness measurements were acquired using a Wilson Rockwell hardness tester. A standard Brale spheroconical diamond penetrator was used with a load of 150 kgf. The hardness readings reported are an average of three measurements. Retained austenite measurements and hardness readings were obtained on the same sample.

The factors and levels selected for the DOE A analysis are shown in Table 1. The well-established heat treatment of 52100 steel[4] was used to aid the selection of the factors and levels shown. A large matrix was selected for the initial DOE to identify all possible interactions between the main factors. Once the interactions between the factors are established for any process, heat treating in this instance, the larger matrix need not be repeated for further refinement of the same process.

The factors and levels for DOE B are shown in Table 2. Three levels were selected for each factor so that any trends in the data would be more readily detected.

The factors and levels for DOE C are shown in Table 3. The factors for the second and third DOEs were the same. The levels for DOE C were selected based upon the results of the second DOE B to further refine the heat-treatment procedure. The range of the factors between levels 1 and 3 was decreased for DOE C.

The factors were assigned to an L16 $(2)^{15}$ array for the first experiment and to an L9 $(3)^4$ orthogonal array for the second and third Taguchi experiments, as shown in Table 4, 5, and 6, respectively. It was assumed that there were no interactions between factors for the second and third experiments. Because it would be difficult and time consuming to heat the coupons individually, the austenitizing temperatures were assigned to

Table 2 Factor and level descriptions for Taguchi DOE B

	Factors	Level 1	Level 2	Level 3
A	Austenitizing temperature	774 °C (1425 °F)	827 °C (1520 °F)	871 °C (1600 °F)
B	Tempering temperature	93 °C (200 °F)	177 °C (350 °F)	343 °C (650 °F)
C	Temper time	1 h	2 h	4 h
D	Cold treatment	None	0.5 h	1 h

Table 3 Factors and level descriptions for Taguchi DOE C

	Factors	Level 1	Level 2	Level 3
A	Austenitizing temperature	774 °C (1425 °F)	802 °C (1475 °F)	827 °C (1520 °F)
B	Tempering temperature	93 °C (200 °F)	135 °C (275 °F)	177 °C (350 °F)
C	Tempering time	1 h	1.5 h	2 h
D	Cold treatment	None	0.25 h	0.5 h

Table 4 $L_{16}^{(2)}$ array for Taguchi DOE A

No.	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O
1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
2	1	1	1	1	1	1	1	2	2	2	2	2	2	2	2
3	1	1	1	2	2	2	2	1	1	1	1	2	2	2	2
4	1	1	1	2	2	2	2	2	2	2	2	1	1	1	1
5	1	2	2	1	1	2	2	1	1	2	2	1	1	2	2
6	1	2	2	1	1	2	2	2	2	1	1	2	2	1	1
7	1	2	2	2	2	1	1	1	1	2	2	2	2	1	1
8	1	2	2	2	2	1	1	2	2	1	1	1	1	2	2
9	2	1	2	1	2	1	2	1	2	1	2	1	2	1	2
10	2	1	2	1	2	1	2	2	1	2	1	2	1	2	1
11	2	1	2	2	1	2	1	1	2	1	2	2	1	2	1
12	2	1	2	2	1	2	1	2	1	2	1	1	2	1	2
13	2	2	2	1	2	2	1	1	2	2	1	1	2	2	1
14	2	2	1	1	2	2	1	2	1	1	2	2	1	1	2
15	2	2	1	2	1	1	2	1	2	2	1	2	1	1	2
16	2	2	1	2	1	1	2	2	1	1	2	1	2	2	1
	1	2			3						4				

Table 5 $L_9\ (3)^4$ Array for Taguchi DOE B

	$L_9(3)^4$				A	B	C	D
Factors Exp.	A 1	B 2	C 3	D 4	Austenitizing temperature	Tempering temperature	Tempering time	Cold treatment
1	1	1	1	1	774 °C (1425 °F)	93 °C (200 °F)	1 h	None
2	1	2	2	2	774 °C (1425 °F)	177 °C (350 °F)	2 h	0.5 h
3	1	3	3	3	774 °C (1425 °F)	343 °C (650 °F)	4 h	1 h
4	2	1	2	3	827 °C (1520 °F)	93 °C (200 °F)	2 h	1 h
5	2	2	3	1	827 °C (1520 °F)	177 °C (350 °F)	4 h	None
6	2	3	1	2	827 °C (1520 °F)	343 °C (650 °F)	1 h	0.5 h
7	3	1	3	2	871 °C (1600 °F)	93 °C (200 °F)	4 h	0.5 h
8	3	2	1	3	871 °C (1600 °F)	177 °C (350 °F)	1 h	1 h
9	3	3	2	1	871 °C (1600 °F)	343 °C (650 °F)	2 h	None

column A1, so that samples could be grouped together during austenitizing. The experiments were then randomized within each group.

The specimens were first austenized at the prescribed temperature for 1.5 h. After reaching the austenitizing temperature, each sample was quenched in oil and was allowed to rest for

Table 6 $L_9(3)^4$ array for Taguchi DOE C

Factors Exp.	$L_9(3)^4$ A 1	B 2	C 3	D 4	A Austenizing temperature	B Tempering temperature	C Tempering time	D Cold treatment
1	1	1	1	1	774 °C (1425 °F)	93 °C (200 °F)	1 h	None
2	1	2	2	2	774 °C (1425 °F)	135 °C (275 °F)	1.5 h	0.25 h
3	1	3	3	3	774 °C (1425 °F)	177 °C (350 °F)	2 h	0.5 h
4	2	1	2	3	802 °C (1475 °F)	93 °C (200 °F)	1.5 h	0.5 h
5	2	2	3	1	802 °C (1475 °F)	135 °C (275 °F)	2 h	None
6	2	3	1	2	802 °C (1475 °F)	177 °C (350 °F)	1 h	0.25 h
7	3	1	3	2	827 °C (1520 °F)	93 °C (200 °F)	2 h	0.25 h
8	3	2	1	3	827 °C (1520 °F)	135 °C (275 °F)	1 h	0.5 h
9	3	3	2	1	827 °C (1520 °F)	177 °C (350 °F)	1.5 h	None

Table 7 Experimental results for Taguchi DOE A

Experiment	Volume percent retained austenite	Hardness (Rockwell C scale)
A1	6.4	59.1
A2	2.8	60.4
A3	7.9	52.9
A4	2.1	53.9
A5	0.2	39.9
A6	0.1	47.8
A7	0.1	38.9
A8	0.1	42.8
A9	5.9	61.8
A10	2.2	62.7
A11	7.2	61.0
A12	1.0	62.1
A13	0	50.6
A14	0	52.7
A15	0	50.2
A16	0	51.3

Table 8 Experimental results for Taguchi DOE B

Experiment	Volume percent retained austenite	Hardness (Rockwell C scale)
B1	15.0	61.1
B2	0	56.6
B3	0	47.9
B4	6.1	65.4
B5	0	58.9
B6	0.1	55.1
B7	10.2	66.7
B8	0	60.9
B9	0	53.2

0.5 h. The cold treatment was then performed using liquid nitrogen for the prescribed amount of time. After the cold treatment and prior to the tempering operation, the samples were again allowed to rest for 0.5 h. The samples that were not cold treated were also allowed to rest for 0.5 h prior to the tempering operation. After tempering, each sample was allowed to cool at room temperature.

Table 9 Experimental results for Taguchi DOE C

Experiment	Volume percent retained austenite	Hardness (Rockwell C scale)
C1	11.5	59.5
C2	2.4	43.5
C3	0	54.0
C4	4.5	62.3
C5	13.4	59.3
C6	0	58.1
C7	6.7	65.0
C8	4.5	62.4
C9	0	58.7

Table 10 Response table for Taguchi DOE A

	Factors	Austenite Level 1	Austenite Level 2	Hardness Level 1	Hardness Level 2
A	Austenitzing temperature	2.5	2.0	49.5	56.6
B	Tempering temperature	4.4	0.1	59.2	46.8
D	Tempering time	2.2	2.6	54.4	51.6
H	Cold treatment	3.5	1.0	51.8	54.2
	Interactions				
C	Austenitization temperature vs tempering temperature	2.4	2.1	53.9	52.1
E	Austenitization temperature vs tempering time	2.2	2.3	54.0	52.0
F	Tempering temperature vs tempering time	2.2	2.3	53.4	52.6
I	Austenitization temperature vs cold treatment	2.2	2.3	52.5	53.6
J	Tempering temperature vs cold treatment	3.5	1.0	53.7	52.3
L	Tempering time vs cold treatment	2.0	2.5	52.7	53.3

3. Results and Discussion

The results obtained for the first (DOE A), second (DOE B), and third (DOE C) experiments are shown in Table 7, 8, and 9, respectively. The retained austenite measurements

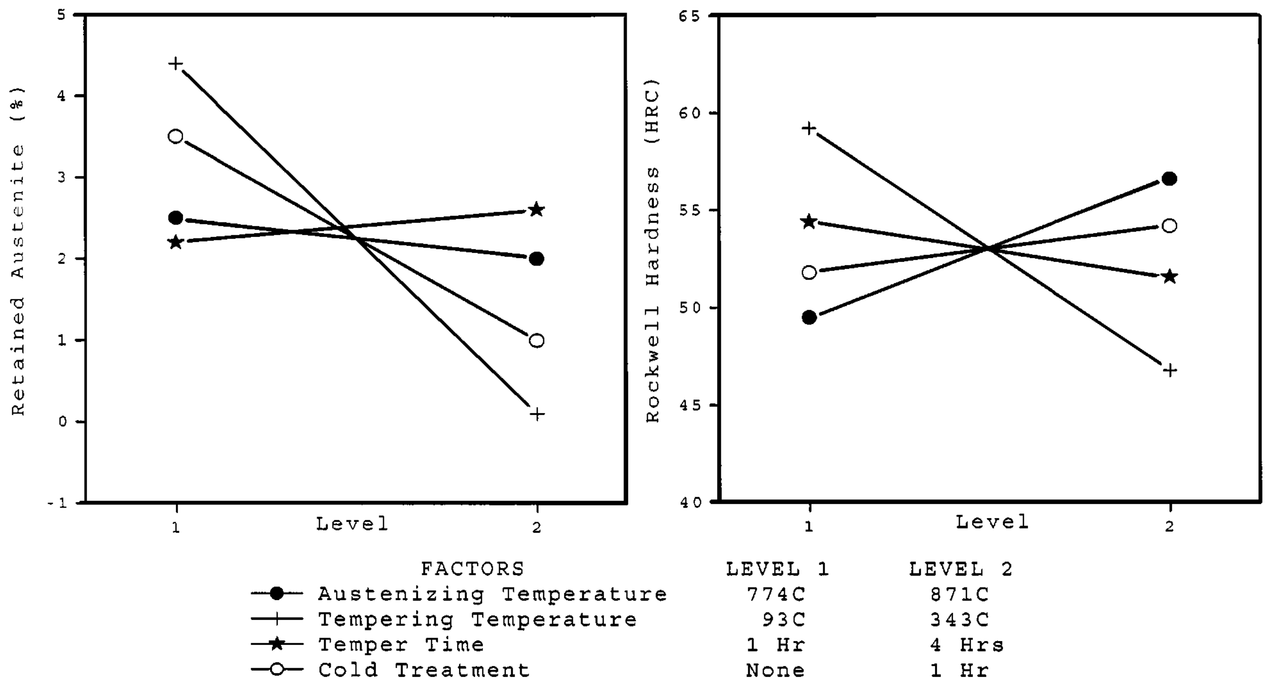


Fig. 1 Plot of response data for main factors of Taguchi DOE A

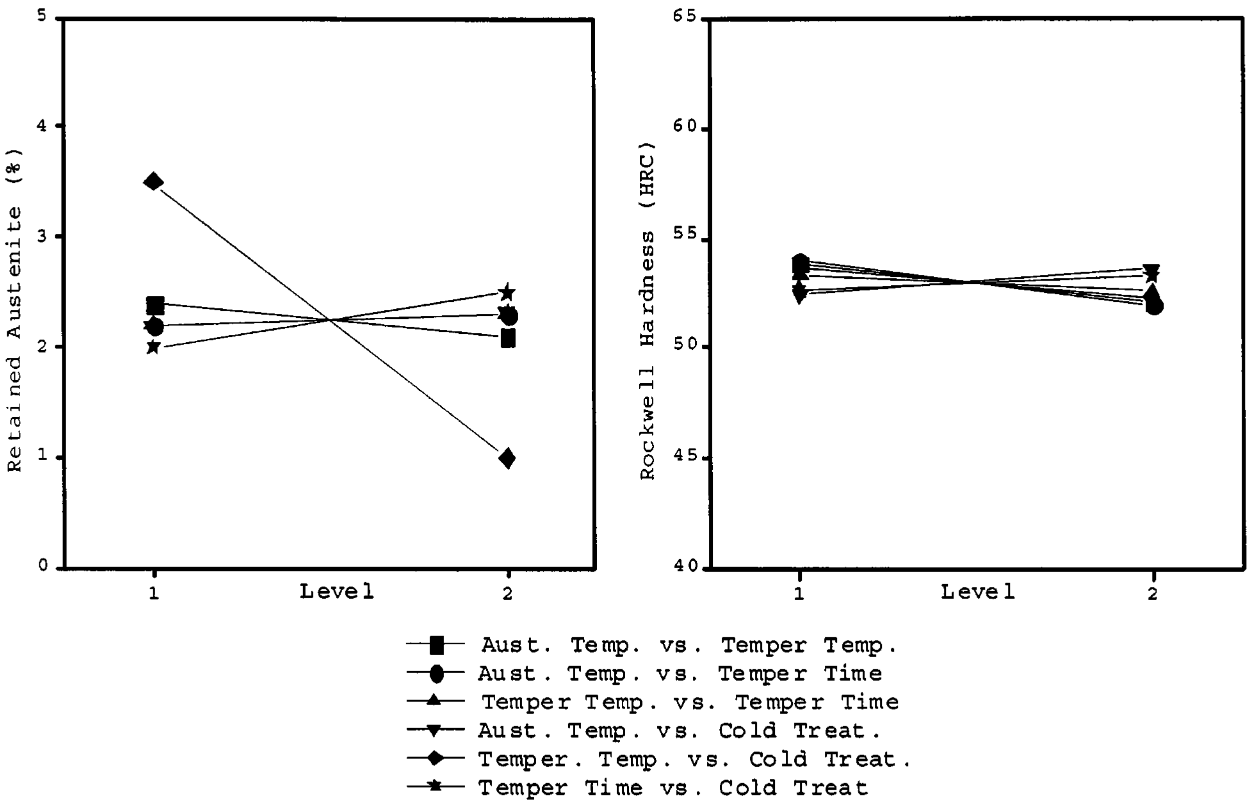


Fig. 2 Plot of response data for interactions of Taguchi DOE A

ranged from 0 to 7.9 vol.% for the first experiment, from 0 to 15% for the second experiment, and from 0 to 13.4% for the third experiment. The Rockwell C hardness ranged from 38 to 63 HRC for the first experiment, from 53 to 67 HRC for the second experiment, and from 44 to 65 HRC for the third experiment. The variation in the data is the result of all of the

Table 11 Response table for Taguchi DOE B

		Austenite			Hardness		
	Factors	Level 1	Level 2	Level 3	Level 1	Level 2	Level 3
A	Austenitizing temperature	5.0	2.1	3.4	55.2	59.8	60.3
B	Tempering temperature	10.4	0	0	64.4	58.8	52.1
C	Tempering time	5.0	2.0	3.4	59.0	58.4	57.8
D	Cold treatment	5.0	3.4	2.0	57.7	59.5	60.0

Table 12 Response table for Taguchi DOE C

		Austenite			Hardness		
	Factors	Level 1	Level 2	Level 3	Level 1	Level 2	Level 3
A	Austenitizing temperature	4.6	6.0	3.7	52.3	59.9	62.0
B	Tempering temperature	7.5	6.8	3.7	62.2	55.1	56.9
C	Tempering time	5.3	2.3	6.7	60.0	54.8	59.4
D	Cold treatment	8.3	3.0	3.0	59.2	55.5	59.6

Table 13 Experimental confirmation

Conditions		
Factors	Condition 1	Condition 2
Austenitizing temperature	827 °C (1520 °F) 177 °C (350 °F) 2 h	827 °C (1520 °F) 177 °C (350 °F)
Cold treatment		None
Results		
Volume percent retained austenite	0	0
Hardness Rockwell C	58.7	57.9

levels (temperatures and times) being different for each set of experiments.

The response data are shown in Table 10 and plotted in Fig. 1 and 2 for the first experiment. The results indicate that the tempering temperature and cold treatment have the most influence and the austenitizing temperature and tempering time have the least influence on the retained austenite levels. The tempering temperature and the austenitizing temperatures appear to have the most influence on the hardness, with the cold treatment and tempering time having some influence. The tempering time and cold treatment seem to be interacting in relation to the retained austenite levels. None of the main factors show strong interactions in relation to hardness. Response data for the second experiment are shown in Table 11 and are plotted in Figure 3. Likewise for the third experiment, response data are shown in Table 12 and are plotted in Figure 4.

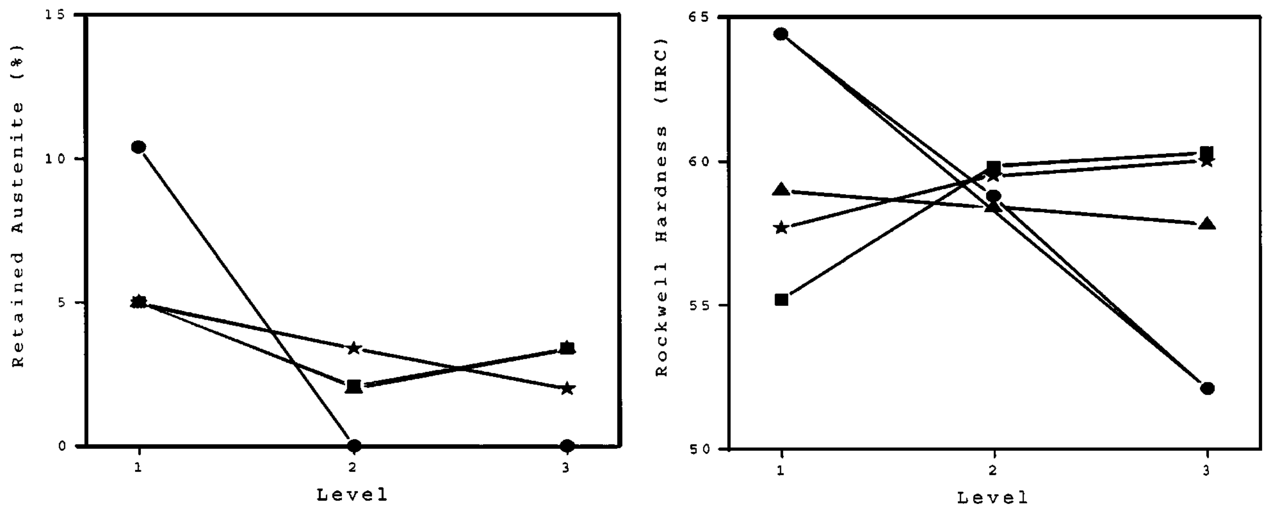
The conditions that gave the lowest austenite content and the highest hardness are shown in Table 13. The results appear to indicate that the cold treatment might have an effect on the hardness of the 52100 steel, but this cannot be confirmed because of the interaction that takes place with the tempering temperature and cold treatment shown in the interactions for DOE A. Therefore, the confirmation experiment was performed under identical conditions with the exception that one sample was cold treated and one sample was not. The confirmation experiment was successful, resulting in no detectable retained austenite and a hardness value on the order of 58 HRC for both samples.

The confirmation results do not substantiate the finding that cold treating may increase the hardness. The confirmation experiment also indicates that, although an interaction exists between the tempering temperature and the cold treatment, the tempering temperature has the most influence on the retained austenite content.

4. Conclusions

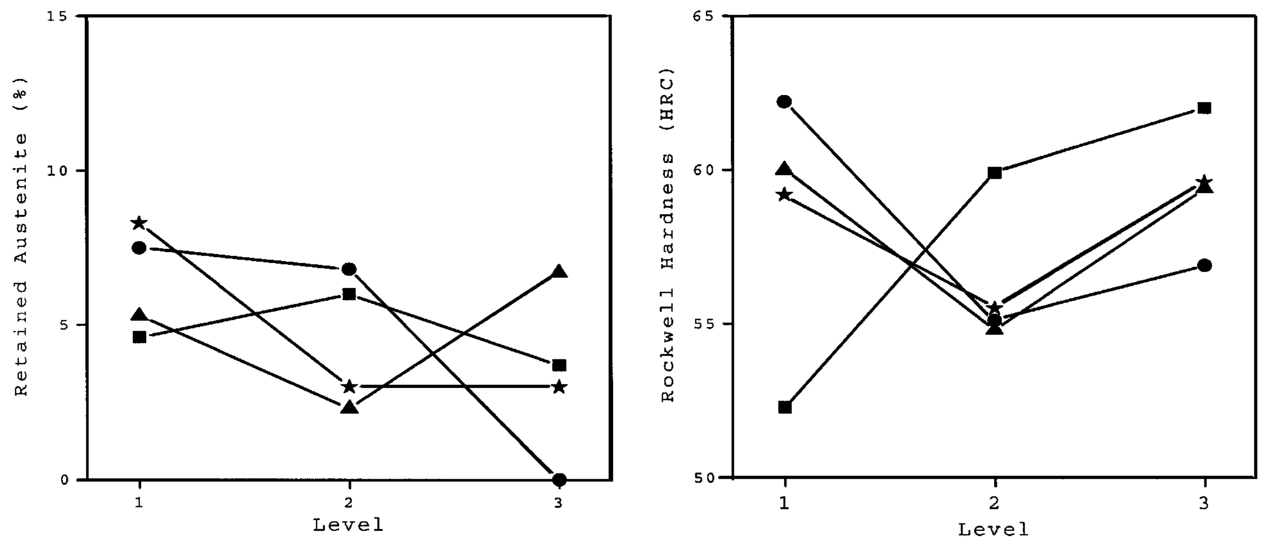
The experiments conducted show that austenitizing and tempering temperatures have the most influence on the retained austenite and the hardness in the heat treatment of 52100 steel. The austenitizing and tempering temperatures of 827 and 177 °C, respectively, gave the lowest austenite and highest hardness values for both the second and final Taguchi analyses, indicating that no further refinement of the experiment is necessary. Therefore, if the goal of heat treating 52100 steel is to produce the lowest austenite content and the highest hardness, either condition 1 or 2, shown in Table 13, could be used. The experiment also indicates that, to produce the best product (low austenite content and high hardness), the process controls should be placed on the austenitizing temperature and the tempering temperature.

This study is intended to illustrate the use of Taguchi DOE methods employing x-ray diffraction retained austenite measurement to efficiently develop heat-treatment parameters for steels. It is not intended to provide optimal parameters for any specific application of 52100 steel. The final heat treatment selected to produce negligible austenite and 58 HRC material is not intended to be optimal for any particular application. However, the same experimental approach can



FACTORS	LEVEL 1	LEVEL 2	LEVEL 3
Austenizing Temperature	774C	877C	871C
Tempering Temperature	93C	177C	343C
Tempering Time	1 Hr	2 Hrs	4 Hrs
Cold Treatment	None	0.5 Hr	1 Hr

Fig. 3 Plot of response data for main factors of DOE B



FACTOR	LEVEL 1	LEVEL 2	LEVEL 3
Austenizing Temperature	774C	802C	827C
Tempering Temperature	93C	135C	177C
Tempering Time	1 Hr	1.5 Hrs	2 Hrs
Cold Treatment	None	0.25 Hr	0.5 Hr

Fig. 4 Plot of response data for main factors of Taguchi DOE C

be used, in principle, to efficiently develop any achievable set of properties in the heat treatment of steels.

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