Improvement of Mechanical Properties of Spheroidized 10B21 Steel Coil Using Taguchi Method of Robust Design

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The development in the fastener industry raises the need for steel wire coils. The wire usually has to be annealed to improve its cold formability. The quality of a spheroidized annealed wire affects the forming quality of screws. In the fastener industry, most companies use a subcritical process for spheroidized annealing. Various parameters affect the quality of spheroidized annealing such as preheating time, spheroidized annealing temperature, prolonged heating time, furnace cooling temperature, and furnace cooling time. The effects of spheroidized annealing parameters affect the quality characteristics of wires, such as tensile strength and ductility. A series of experimental tests on AISI 10B21 steel wires is carried out in a vacuum drying oven and the Taguchi method of robust design is used to obtain optimal spheroidized annealing conditions to improve the mechanical properties of steel wires for cold forming. The experimental results show that the tensile strength is the main quality characteristic of spheroidized annealed steel wires, and that the spheroidized annealing temperature and prolonged heating time have the greatest effect on the mechanical properties of AISI 10B21 steel wires. A comparison between the results obtained using the optimal spheroidizing conditions and the measures determined using the original settings shows that the new spheroidizing parameter settings effectively improve the performance measures over their values at the original settings. The formability of AISI 10B21 steel wires is effectively improved.

1. Introduction

The spheroidized annealing treatment of steel wire coils affects the cold formability for manufacturing fasteners. The wire is generally produced by drawing a wire coil to a wire, which requires softening before cold heading.¹,² With increasing motion of dislocation, the yield strength, tensile strength, and hardness increase, but ductility reaches zero.³ Once it is deformed continuously until the yield point is passed, the material will exhibit brittle fracture. To improve the cold formability and mechanical properties of the wire, the drawn wire must

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go through annealing. By removing the effects of internal stress and enhancing ductility, the cold formability is increased and the forming impact force is reduced.

Many studies on the mechanisms and kinetics of spheroidization were proposed. Tian and Kraft developed an early theorization indicating that spheroidization is associated with morphological defects such as kinetics acceleration. O’Brien and Hosford investigated the spheroidization of the medium carbon steels, AISI 1541 and AISI 4037, used in the bolt industry with two process cycles, namely, intercritical and subcritical cycles. Introducing defects into cementites by severe plastic deformation is one of the effective methods for increasing the spheroidization speed. Hono et al. revealed that cementites in a near eutectic steel spheroidize more easily after a severe drawing. Shin et al. studied the enhanced spheroidization kinetics in terms of carbon dissolution from cementites and defects induced in cementites by a severe plastic deformation, and revealed that the increase in accumulated strain in the equal-channel angular pressed steel decreased the spheroidization temperature and time. Gul’ et al. developed a new method for a more intense spheroidization of cementites to accelerate spheroidization. Spheroidization is induced by nonisothermal holding at high temperatures using an internal heat source. An empirical research study demonstrates that the Taguchi method is useful for improving the quality of an AISI 1022 steel wire for cold heading. The results show that the spheroidized annealing temperature and prolonged heating time have the greatest effect on the mechanical properties of steel wires. Ko et al. proposed a method of continuous shear drawing (CSD) for industrial applications to steel wire manufacturing and compared the spheroidization behavior of medium-carbon steel processed by CSD to that processed by conventional drawing. The shear drawing process obviously affected the acceleration of spheroidization of the wire through a large amount of plastic deformation as compared with the wire drawing process. Min and Ha conducted the spheroidization heat treatment of SK85 high-carbon steel sheets with various initial microstructures obtained after cold rolling at various reduction ratios at two annealing temperatures. As spheroidized annealing proceeded, the fragmentation of cementite plates, the spheroidization of cementite platelets, and coarsening were observed consecutively. The elongation of cold-rolled specimens markedly increases with spheroidized annealing time. Joo et al. indicated that the cementite in steel spheroidizes much more easily after a severe drawing, and SEM results also revealed that the prior cold working could increase the spheroidization ratio with cold workability improved by subcritical annealing.

In the bolt industry, most companies used a subcritical process for spheroidized annealing, simply heating to a temperature below the lower critical and holding. The cold heading quality of the AISI 10B21 steel wire is usually used to manufacture welding bolts and flange weld nuts for automotive fasteners. The wire must be spheroidized annealed after drawing the wire coil (⌀6.5 mm) to a specific size with section-area reductions of about 20.1%. The quality of the spheroidized annealed wire affects the forming quality of screws. Various parameters affect the quality characteristics of spheroidized annealing, such as preheating time, spheroidized annealing temperature, prolonged heating time, furnace cooling temperature, and furnace cooling time. The effects of spheroidized annealing parameters affect the quality characteristics of wires, such as tensile strength, ductility, and hardness. In this study, a series of experimental
tests on AISI 10B21 steel wires is carried out in a vacuum drying oven and the Taguchi method is used to obtain optimal spheroidized annealing conditions to improve the mechanical properties of steel wires for cold forming.

2. Experimental Design

In this study, a subcritical process is used for the spheroidized annealing of steel wires, simply heating them to a temperature below the lower critical temperature and holding at this temperature. A series of experimental tests on AISI 10B21 low-carbon steel wires is carried out in a vacuum drying oven. The five process parameters with four levels listed in Table 1 are chosen as the experimental factors in this study. Every factor has four levels to spheroidize wires in order to evaluate the mechanical properties of wires. The parameters of Level 3 are the original spheroidized annealing process conditions, which are used in Jinn Her Enterprise Co., Ltd., Taiwan.

The Taguchi method allows simultaneous changes of many factors in a systematic manner, ensuring the reliable and independent study of the effects of such factors. The orthogonal array table, L₁₆(4⁵),\(^{13,14}\) is used as an experimental design for five factors and is shown in Table 2.

| Table 1 | Experimental factors and their levels for L₁₆ orthogonal array. |
|---------|---------------------------------------------------------------|
| Factor | Level 1 | Level 2 | Level 3 | Level 4 |
| A: Preheating time (h) | 4 | 5 | 6 | 7 |
| B: Spheroidized annealing temperature (°C) | 680 | 705 | 720 | 725 |
| C: Prolonged heating time (h) | 8 | 7 | 6 | 5 |
| D: Furnace cooling temperature (°C) | 550 | 500 | 450 | 400 |
| E: Furnace cooling time (h) | 5 | 8 | 10 | 11 |

| Table 2 | L₁₆(4⁵) orthogonal array for experimental parameter assignment. |
|---------|----------------------------------------------------------------|
| Exp. No. | A: Preheating time (h) | B: Spheroidized annealing temperature (°C) | C: Prolonged heating time (h) | D: Furnace cooling temperature (°C) | E: Furnace cooling time (h) |
| L1 | 4 | 680 | 8 | 550 | 5 |
| L2 | 4 | 705 | 7 | 500 | 8 |
| L3 | 4 | 720 | 6 | 450 | 10 |
| L4 | 4 | 725 | 5 | 400 | 11 |
| L5 | 5 | 680 | 7 | 450 | 11 |
| L6 | 5 | 705 | 8 | 400 | 10 |
| L7 | 5 | 720 | 5 | 550 | 8 |
| L8 | 5 | 725 | 6 | 500 | 5 |
| L9 | 6 | 680 | 6 | 400 | 8 |
| L10 | 6 | 705 | 5 | 450 | 5 |
| L11 | 6 | 720 | 8 | 500 | 11 |
| L12 | 6 | 725 | 7 | 550 | 10 |
| L13 | 7 | 680 | 5 | 500 | 10 |
| L14 | 7 | 705 | 6 | 550 | 11 |
| L15 | 7 | 720 | 7 | 400 | 5 |
| L16 | 7 | 725 | 8 | 450 | 8 |
Two quality characteristics of the spheroidized annealed wire, namely, tensile strength and ductility, are investigated. Each test trial, including six specimens, is followed by a fabrication process and the results are transformed to signal-to-noise (S/N) ratios. Spheroidizing provides the needed ductility for cold heading. The ductility of the steel wire may be improved through spheroidized annealing and their hardness may be reduced as well. The tensile test is used as a measure of ductility by calculating the elongation of the specimen upon fracture.\(^{(15)}\) Therefore, in terms of the desired characteristics for ductility, the higher the better, and the S/N ratio is\(^{(14)}\)

\[
S/N = -10 \log \frac{\sum_{i=1}^{n} 1/y_i^2}{n},
\]

where \(y_i\) is the ductility (elongation, \(\varepsilon_f\)) of each specimen and \(n\) is the test number.

When the ductility of the steel wire is improved through spheroidized annealing, the strength of the steel wire is simultaneously decreased. However, the given strength of the annealed steel wire must be provided for cold heading. Therefore, the tensile strength of the steel wire is the main quality characteristic, with a target value of 402 MPa, which is assigned by Jinn Her Enterprise Co., Ltd., Taiwan. The S/N ratio for the nominal-the-best response is\(^{(14)}\)

\[
S/N = -10 \log [(\mu - m)^2 + S^2],
\]

where \(\mu\) is the mean of each trial, \(m\) is the target value, and \(S\) is the standard deviation. Tensile tests are conducted on a 20 ton universal testing machine under a constant ram speed of 5 mm/min at room temperature. The dimensions of the tensile specimen are \(\Theta 5.81\) mm × \(L 200\) mm.

The analysis of variance (ANOVA) is an effective method of determining the significant factors and optimal fabrication conditions required to obtain the optimal quality. For the Taguchi method, the experimental error is evaluated using ANOVA to test the significance of various factors. The nature of the interaction between factors is considered as the experimental error.\(^{(14)}\) If the effect of a factor in comparison with the experimental error is sufficiently large, it is identified as a significant factor. The confidence level of a factor is evaluated using the experimental error to identify the significant factor that affects the material properties of steel wires.

3. Materials and Methods

In this study, the wire is spheroidized annealed after drawing the AISI 10B21 steel wire coil (\(\Theta 6.5\) mm) to a specific size (\(\Theta 5.81\) mm) with a section-area reduction of about 20.1%. The steel wire coil is manufactured (\(\Theta 6.5\) mm, Al-killed) by China Steel Corporation, Kaohsiung, Taiwan. Its chemical composition is shown in Table 3.

The steel wire is spheroidized annealed, as shown in Fig. 1, with a CVM-20S vacuum drying oven (CHENG SANG Scientific Changhua, Taiwan). The Taguchi method allows the simultaneous changes of many factors in a systematic manner. The orthogonal array table, \(L_{16}(4^5)\), is used as an experimental design for the factors,\(^{(14)}\) as shown in Table 1.
4. Results and Discussion

When the wire is produced by drawing a wire coil to a wire, the tensile strength, ductility, and hardness are respectively about 635 MPa, 0.09, and 94.5 HRB owing to heavy plastic work. Spheroidization is the process of producing a microstructure in which the cementite exhibits a spheroidal distribution. The obtained globular structure improves the formability of the steel wire. When the wire is fabricated following the original spheroidized annealing conditions (Level 3 in Table 1), the mean tensile strength, mean ductility, and mean hardness are 369.6 MPa, 0.42, and 64.3 HRB, respectively.

The experimental results of the tensile strength and ductility (mean, $\mu$; standard deviation, $S$; and $S/N$ ratio) of the spheroidized annealed steel wire are shown in Tables 4 and 5, respectively. The mean tensile strength varies from 360.7 to 382.7 MPa and is less than the target value, as shown in Table 4. The mean ductility of test L13 is the largest and close to the target value. The standard deviation varies from 1.34 to 5.28 MPa and test L5 is the smallest among the sixteen tests.

As shown in Table 5, the mean ductility varies from 0.37 to 0.45, and the mean values of tests L3, L4, L8, L11, and L12 are larger than the value at the original settings. The standard deviation varies from 0.009 to 0.038. The standard deviations of tests L9 and L13 are the smallest among the sixteen tests. The properties of the spheroidized annealed steel wire are obviously varied under various spheroidized annealing process conditions.

4.1 Tensile strength

To obtain the optimal quality, ANOVA is carried out to determine significant factors and optimal fabrication conditions. The contribution and confidence level of each factor constructed in Table 6 could identify the significant factor affecting the tensile strength of the wire. The

![Table 3](52.png)

Table 3
Chemical composition of AISI 10B21 low-carbon steel wires (wt%).

| C   | Mn  | P     | S     | Si   | Al   | B     |
|-----|-----|-------|-------|------|------|-------|
| 0.20–0.21 | 0.80–0.83 | 0.015–0.019 | 0.005–0.007 | 0.04–0.06 | 0.038–0.051 | 0.0020–0.0021 |

![Fig. 1](53.png)

Fig. 1. (Color online) Spheroidized annealing procedure.
contribution of a factor is the percentage of the sum of squares (SS), that is, the percentage of the factor variance to the total quality loss.\(^{13,14}\) The effect of a factor may be pooled to error if its confidence level or contribution is relatively small. It is obvious from the ANOVA table that the contribution of spheroidized annealing temperature (B) is 79.7% of the total variation, which is obviously the highest contributor to the variability of the experimental results. The contribution of prolonged heating time (C) is 12.2%, which is the second highest contribution.
However, the other three factors are not significant for the S/N ratio since their contributions are relatively small. With the pooling of errors from the nonsignificant factors (A, D, and E), the error for the S/N ratio is estimated\(^\text{(14)}\) and then the confidence levels are 100.0 and 96.5%, respectively, for spheroidized annealing temperature (B) and prolonged heating time (C). That is, both factors, particularly the spheroidized annealing temperature, significantly affect the tensile strength of the steel wire with a confidence level of more than 95.0%.

Figure 2 illustrates the factor response diagram and the level averages of five factors with respect to the S/N ratio. For each factor, the effect is indicated by the range of level averages and the maximum level average is considered the optimal level.\(^\text{(13,14)}\) It is obviously revealed that, for the five factors, the original levels (Level 3) are not the optimal fabrication parameters for obtaining the target tensile strength. For the significant factors of spheroidized annealing temperature (B) and prolonged heating time (C), Level 1 for the spheroidized annealing temperature (680 °C, B1) and Level 4 for the prolonged heating time (5 h, C4) are evidently the optimal levels, as shown in Fig. 2. However, it is observed that the response is not linear with the annealing temperature, but is almost linear with the prolonged heating time. For the spheroidized annealing temperature, the response of the optimal level is much more effective than the other three levels. The effects of the preheating time (A), furnace cooling temperature (D), and furnace cooling time (E) are relatively small. The optimal levels are Level 2 for the preheating time (5 h, A2), Level 3 for the furnace cooling temperature (450 °C, D3), and Level 1 for the furnace cooling time (5 h, E1).

### 4.2 Ductility

For the ductility of the annealed steel wire, the ANOVA results of the S/N ratio are shown in Table 7. It is evident from Table 7 that the highest contributor to the variability

| Table 6 | ANOVA results of S/N ratio for tensile strength. |
|---------|----------------------------------|
| Factor  | SS     | DOF | Var. | Contribution (%) |
| A       | 0.58   | 3   | 0.19 | 0.9              |
| B       | 50.60  | 3   | 16.87| 79.7             |
| C       | 7.73   | 3   | 2.58 | 12.2             |
| D       | 1.20   | 3   | 0.40 | 1.9              |
| E       | 3.39   | 3   | 1.13 | 5.3              |
| Total   | 63.50  | 15  | —    | 100.0            |

**Pooling of errors**

| Factor  | SS     | DOF | Var. | F   | Confidence (%) | Significance |
|---------|--------|-----|------|-----|----------------|--------------|
| A       |        |     |      |     |                |              |
| B       | 50.60  | 3   | 16.87| 29.38| 100.0          | Yes          |
| C       | 7.73   | 3   | 2.58 | 4.49 | 96.5           | Yes          |
| D       | Pooled |     |      |     |                |              |
| E       | Pooled |     |      |     |                |              |
| Error   | 5.17   | 9   | 0.57 |     | Sexp = 0.76    |              |
| Total   | 63.50  | 15  | —    |     | *At least 95.0% confidence level | |

SS, sum of squares; DOF, degree of freedom; Var., variance; F, F-ratio; Sexp, experimental error.
of the experimental results is also the spheroidized annealing temperature (B, 63.8%). The contribution of preheating time (A) is 18.1%, which is the second highest contribution. However, the prolonged heating time (C), furnace cooling temperature (D), and furnace cooling time (E) are not significant factors because their contributions are relatively small. With the pooling of errors from the nonsignificant factors (C, D, and E), the confidence levels are 91.1 and 99.7%, respectively, for the preheating time (A) and spheroidized annealing temperature (B). That is, the ductility of the steel wire is significantly affected by the preheating time and spheroidized annealing temperature, with a confidence level of more than 90.0%.

The factor response diagram and the level averages of the five factors with respect to the 

![Factor response diagram for tensile strength.](image)

**Table 7**

| Factor | SS  | DOF | Var. | Contribution (%) |
|--------|-----|-----|------|------------------|
| A      | 0.71| 3   | 0.24 | 18.1             |
| B      | 2.52| 3   | 0.84 | 63.8             |
| C      | 0.32| 3   | 0.11 | 8.2              |
| D      | 0.12| 3   | 0.04 | 2.9              |
| E      | 0.28| 3   | 0.09 | 7.0              |
| Total  | 3.95| 15  |      | 100.0            |

Pooling of errors

| Factor | SS  | DOF | Var. | F  | Confidence (%) | Significance |
|--------|-----|-----|------|----|----------------|--------------|
| A      | 0.71| 3   | 0.24 | 2.99| 91.1           | Yes          |
| B      | 2.52| 3   | 0.84 | 10.53| 99.7           | Yes          |
| C      |     |     |      |    | Pooled         |              |
| D      |     |     |      |    | Pooled         |              |
| E      |     |     |      |    | Pooled         |              |
| Error  | 0.72| 9   | 0.08 |    | $S_{exp} = 0.28$ |              |
| Total  | 3.95| 15  |      |    | *At least 90.0% confidence level |              |

SS, sum of squares; DOF, degree of freedom; Var., variance; F, F-ratio; $S_{exp}$, experimental error
time (C), furnace cooling temperature (D), and furnace cooling time (E) are relatively small. The optimal levels are Level 3 for the prolonged heating time (6 h, C3), Level 1 for the furnace cooling temperature (550 °C, D1), and Level 3 for the furnace cooling time (10 h, E3).

With the results of the analysis of the optimal quality characteristics of tensile strength and ductility, the optimal conditions are shown in Table 8. The spheroidized annealing temperature (B) is obviously significant for both tensile strength and ductility but at different levels. Since the tensile strength of the steel wire is the main quality characteristic and has a higher effect, the optimal level is thus determined as Level 1 for the spheroidized annealing temperature (680 °C, B1). The prolonged heating time (C) is significant for the tensile strength but not for ductility. Thus, Level 4 for the prolonged heating time (5 h, C4) is then chosen as the optimal level. However, the preheating time (A) is not significant for the tensile strength, but is significant for the ductility. Level 3 for the preheating time (6 h, A3) is chosen as the optimal level. The factors of furnace cooling temperature (D) and furnace cooling time (E) are not significant either for the tensile strength or ductility. The original level, Level 3, for the furnace cooling temperature (450 °C, D3) and furnace cooling time (10 h, E3) is determined.

4.3 Confirmation experiments

To verify the predicted results, the steel wire is fabricated using the optimal levels A3, B1, C4, D3, and E3, as described in Table 8. Figures 4 and 5 show the nontreated, original (using Level 3s in Table 1), and optimal probability distributions for the tensile strength and ductility of the steel wire, respectively.

It is observed that, through spheroidized annealing with the original settings, the mean tensile strength decreases substantially by about 265 MPa as compared with the nonspheroidized results and the deviation decreases, as shown in Fig. 4. Simultaneously, the mean ductility markedly increases by about 0.33, but the deviation increases over six times, as shown in Fig. 5. The formability of the steel wire is much improved, but the strength is insufficient.

Compared with the original results, the optimal mean tensile strength of 388.6 MPa increases and becomes closer to the target value, as shown in Fig. 4, while the deviation slightly increases. The optimal mean ductility of 0.418, as shown in Fig. 5, is almost the same as the original mean ductility of 0.420. However, the deviation obviously decreases by about 76% as compared with
The new parameter settings evidently improve the performance measures, such as strength and ductility, over their values at the original settings, as well as the quality of the spheroidized annealed steel wire. Therefore, the formability of the AISI 10B21 steel wire is effectively improved.

Table 8
Optimal conditions for spheroidized annealing.

| Factor | Tensile strength | Ductility | Optimal |
|--------|-----------------|-----------|---------|
| A: Preheating time (h) | A2 | A3<sup>*</sup> | A3 |
| B: Spheroidized annealing temperature (°C) | B1<sup>*</sup> | B4<sup>*</sup> | B1 |
| C: Prolonged heating time (h) | C4<sup>*</sup> | C3 | C4 |
| D: Furnace cooling temperature (°C) | D3 | D1 | D3 |
| E: Furnace cooling time (h) | E1 | E3 | E3 |

<sup>*</sup>Significant factor

![Fig. 4](Color online) Probability distribution diagram for tensile strength.

![Fig. 5](Color online) Probability distribution diagram for ductility.

the original result. The new parameter settings evidently improve the performance measures, such as strength and ductility, over their values at the original settings, as well as the quality of the spheroidized annealed steel wire. Therefore, the formability of the AISI 10B21 steel wire is effectively improved.

5. Conclusions

The majority of spheroidizing activities are performed to improve the cold formability of steel wires. The spheroidized annealing treatment of steel wire coils affects the cold formability for manufacturing fasteners. The quality of spheroidized annealed steel wires affects the
forming quality of screws. In this study, the Taguchi method is used to obtain optimal spheroidized annealing conditions to improve the mechanical properties of AISI 10B21 steel wires. The spheroidized annealing qualities of steel wires are affected by various factors, such as the preheating time, spheroidized annealing temperature, prolonged heating time, furnace cooling temperature, and furnace cooling time. The spheroidized annealing conditions affect the quality characteristics of steel wires, such as tensile strength and ductility. Since the given strength of the annealing steel wire must be provided for cold heading, the tensile strength is the main quality characteristic of spheroidized annealed steel wires, with a target value of 402 MPa. It is experimentally revealed that the spheroidized annealing temperature (B) and prolonged heating time (C) are significant factors; the determined levels are Level 1 for the spheroidized annealing temperature (680 °C, B1), Level 4 for the prolonged heating time (5 h, C4), Level 3 for the preheating time (6 h, A3), Level 3 for the furnace cooling temperature (450 °C, D3), and Level 3 for the furnace cooling time (10 h, E3). Therefore, the optimal mean tensile strength of 388.6 MPa and the optimal mean ductility of 0.418 are obtained. The new spheroidizing parameter settings evidently improve the performance measures over their values at the original settings. The formability of AISI 10B21 steel wires is effectively improved. The results may be used as a reference for wire manufacturers.

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