OPTIMISING THE PARAMETERS AND INCREASING THE HARDNESS OF DUPLEX STAINLESS STEEL BY ANNEALING PROCESS IN VALVE MANUFACTURING INDUSTRIES

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Abstract: Duplex stainless steels (DSS) are designed to have a phase assemblage consisting of approximately 50% austenite and 50% ferrite. Such an assemblage makes these materials very attractive by providing an excellent combination of high hardness, toughness, and corrosion resistance. This project discuss about the increasing of hardness in duplex stainless steel (DSS), by suitable annealing process. Increasing of austenite increases the hardness of the material and the increasing of ferrite increases the ductile nature of the material. It is too crucial to obtain the suitable result by suitable match of annealing process. Thus the Design of Experiments were done and heating temperature of 1240°C with a holding time of 4.5hrs and cooled to a temperature of 1043.9°Cprovides the maximum hardness value of 311.235 BHN.

1. Introduction

The presence of austenite content in the material is responsible for hardness of the material, so the study had been done to increase the austenite content of the material. Heating temperature, cooling temperature, holding time is the three major parameters which alter the hardness when increased or decreased. A cumulative value with the increased hardness is to be found by using DOE. Annealing heat treatment method changes chemical and sometimes physical properties of a material to decrease the ductility nature and developing the hardness of the material, enhances to work longer. It is reason to heat the material above the recrystallization temperature and maintaining a suitable amount of time with suitable temperature, then cooling it into the required temperature. Heating temperature, cooling temperature and holding time are important process parameters for obtaining fine microstructure. Several articles have recommended that the steels should be heated into the temperature region between the upper and lower critical temperatures to get a fine microstructure. Parametric constraints for annealing (ASTM A995/A995M-Gr.5A (CE3MN)), the references were studied to determine the influenced process parameter and their ranges. Paramount mechanical properties can be obtained by controlling the influenced process parameters. The influenced parameters like Heating temperature, Holding time and cooling temperature were varied while the other input variables were kept constant. Based on this concept the experimental methodologies were done and the results were discussed below.

2. Experimental methodology

Based on the journals referred and experiments conducted, it was observed that the heating temperature above 1140°C have appreciable improvement in the mechanical properties due to needle ferrite formation hence, heating temperature above 1140°C is taken into account. Lower shot velocity results in porosities, whereas increased holding time also leads to improvement of hardness. Therefore, holding time from 3.5 to 4.5hrs was taken into account. Cooling temperature, when the specimen is heated in the furnace there will be a austenite in the specimen so when the cooling is done there will be a formation of the ferrite content which gives ductility to the material so when the cooling temperature is increased above 1045°C there will be a less ferrite formation, which results in a increased hardness.
The process parameter range considered for conducting the experiments are listed below,

Heating temperature: \( 1090 \leq 1140 \geq 1240 \)

Holding time: \( 3.5 \leq 4 \geq 4.5 \)

Cooling temperature: \( 995 \leq 1045 \geq 1095 \)

The structure of the selected design of experiments should be such that the experimental investigation complies with full factorial design. This means that every possible combination between the levels of the various factors is analyzed. For the \( 3^3 \) full factorial design, heating temperature, holding time and cooling temperature were selected as factors, and each was run at three levels and the experiments were carried out by varying the process parameter based on the full fractional approach. Heating temperature, holding time and cooling temperature are the major three input parameters which are taken into account for this project work. The process parameters and the change in hardness according to the change in process parameters were studied and their changes are plotted in the graph.

2.1 Heating Temperature v/s Hardness

![Temperature vs Hardness](image1)

Figure 1. Heating Temperature v/s Hardness

2.2 Hardness v/s Time

![Hardness vs Time](image2)

![Ferrite vs Time](image3)

Figure 2. Hardness v/s Time
2.3 Cooling Temperature v/s Time

![Ferrite vs Cooling Temperature](image1.png)

![Hardness vs Cooling Temperature](image2.png)

**Figure 3.** Cooling Temperature v/s Time

| Std | Run | Factor 1          | Factor 2 | Factor 3          | Response 1 |
|-----|-----|-------------------|----------|-------------------|------------|
|     |     | A: Heating temperature | B: Holding time | C: Cooling time | Hardness |
|     |     | DEGREE (°)         | hrs      | DEGREE (°)        | BHN        |
| 4   | 7   | 1190              | 4        | 1045              | 276        |
| 5   | 11  | 1190              | 4        | 1045              | 278        |
| 3   | 12  | 1190              | 3.5      | 1045              | 276        |
| 14  | 1   | 1190              | 4        | 1045              | 278        |
| 12  | 2   | 1240              | 4        | 1045              | 290        |
| 7   | 3   | 1240              | 4.5      | 995               | 304        |
| 11  | 4   | 1140              | 3.5      | 995               | 260        |
| 8   | 8   | 1190              | 4.5      | 1045              | 291        |
| 15  | 9   | 1190              | 4        | 1045              | 279        |
| 9   | 10  | 1190              | 4        | 995               | 261        |
| 13  | 14  | 1140              | 4        | 1045              | 275        |
| 10  | 15  | 1140              | 4.5      | 1095              | 285        |
| 6   | 5   | 1240              | 3.5      | 1095              | 301        |
| 1   | 6   | 1190              | 4        | 1045              | 280        |
| 2   | 13  | 1190              | 4        | 1095              | 282        |

**Table 1.** Brinell Hardness Value
By collaborating the above values, different combination of parameters were obtained and according to the parameters, hardness test is conducted in brinell hardness machine and the best 15 results are plotted above. According to the hardness value obtained from the brinell hardness machine and by the predicted data on the hardness and according to the change in the process parameters the DOE will be conducted.

3. Results and Discussion

3.1 Statistical Analyses

The Table 3.1 statistical analysis shows that F-values attained 112.5, 112.5 and 220.5 for heating temperature, holding time and cooling time respectively. The parameters attained highest value will influence the responses utmost. From the Table 1, it has been observed that the F statistical value attained about 44.34 for cooling time. From the statistical analysis, it is confirmed that cooling time influences the hardness utmost compare to heating temperature and holding time.

Table 2. Statistical Analyses for Hardness

| Parameters               | Sum of  | df | Mean    | F-value   | p-value  |
|--------------------------|---------|----|---------|-----------|----------|
| A-HEATING TEMPERATURE    | 112.5   | 1  | 112.5   | 22.62421  | 0.005075 |
| B-HOLDING TIME           | 112.5   | 1  | 112.5   | 22.62421  | 0.005075 |
| C-COOLING TIME           | 220.5   | 1  | 220.5   | 44.34345  | 0.001152 |
| AB                       | 33.3333 | 1  | 33.3333 | 6.70347   | 0.04889  |
| AC                       | 0.33333 | 1  | 0.33333 | 0.067035  | 0.806025 |
| BC                       | 75      | 1  | 75      | 15.08281  | 0.011599 |
| A²                       | 112.509 | 1  | 112.509 | 22.62603  | 0.005074 |
| B²                       | 149.4321| 1  | 149.4321| 30.05141  | 0.002755 |
| C²                       | 51.58597| 1  | 51.58597| 10.37415  | 0.023442 |

**HARDNESS** = +2024.95049 - 7.10300 Heating Temperature - 151.38235 Holding Time + 4.88482 Cooling Time + 0.200000 Heating Temperature * Holding Time + 0.000200 Heating Temperature * Cooling Time - 0.300000 Holding Time * Cooling Time + 0.002624 Heating Temperature² + 30.23529 Holding Time² - 0.001776 Cooling Time³.

3.2 Interactive and direct effects of Heating temperature, Holding Time and cooling temperature on annealing process

The effect of each parameter on responses can be analyzed graphically using the interaction surface plots. It shows the relationship of process parameters on responses in way of linear and non-linear function. The interaction plots helps to understand the effects of variation considering one process parameter simultaneously with constant other two parameter. The interaction and directs effect figures obtained for the response hardness is shown in...
Figures 6.1(a-c). There are several observations made from the interaction direct effect figures are discussed in the following. It has been observed that increase in heating temperature increases the hardness drastically because, the metal after heating above 1140°C there will be the needle ferrite formation while cooling which results in the increase of hardness. While considering the cooling temperature, the metal which is heated to a certain temperature will be 100 percent austenite and while cooling there will be the formation of ferrite content. So while increasing the cooling temperature above 1045°C there will be less ferrite which increases the hardness of the material. However, an F- statistical value reveals that the process parameters holding time and cooling temperature also have a considerable influence on hardness. At maximum heating temperature, the improved results can be obtained by using the following. Mechanical properties are influenced by the increased heating temperature with holding time and cooling temperature. From the table below DOE for the different combination is obtained, from this the heating temperature of 1240°C with holding time of 4.5hrs and cooling temperature of 1040.3°C yields the maximum results of 310.9483BHN with a desirability of 0.986.

Table 3. Result Analyses for Hardness

| Number | Heating temperature | Holding time | Cooling time | Hardness | Desirability | Desirability |
|--------|---------------------|--------------|--------------|----------|--------------|--------------|
| 1      | 1240                | 4.5          | 1040.333     | 310.9483 | 0.986032     | 1 Selected   |
| 2      | 1240                | 4.5          | 1048.635     | 311.5447 | 0.985673     | 1            |
| 3      | 1240                | 4.498528     | 1044.663     | 311.2085 | 0.985217     | 1            |
| 4      | 1239.854            | 4.499998     | 1046.422     | 311.335  | 0.985119     | 1            |
| 5      | 1240                | 4.5          | 1037.727     | 310.7107 | 0.984954     | 1            |
| 6      | 1240                | 4.499999     | 1050.451     | 311.6424 | 0.984854     | 1            |
| 7      | 1239.569            | 4.499993     | 1044.501     | 311.0577 | 0.982999     | 1            |
| 8      | 1240                | 4.5          | 1054.185     | 311.8068 | 0.982365     | 1            |
| 9      | 1240                | 4.495273     | 1042.704     | 310.8799 | 0.982153     | 1            |
| 10     | 1240                | 4.495153     | 1045.614     | 311.0898 | 0.982106     | 1            |
| 11     | 1240                | 4.494442     | 1048.682     | 311.2474 | 0.980814     | 1            |
| 12     | 1239.39             | 4.5          | 1039.45      | 310.5594 | 0.980622     | 1            |
| 13     | 1240                | 4.498445     | 1055.221     | 311.7625 | 0.980212     | 1            |
| 14     | 1240                | 4.5          | 1057.455     | 311.91   | 0.979309     | 1            |
| 15     | 1239.047            | 4.499999     | 1046.89      | 310.9531 | 0.978389     | 1            |
| 16     | 1240                | 4.490207     | 1047.967     | 310.9764 | 0.97733      | 1            |
| 17     | 1240                | 4.499999     | 1029.349     | 309.7829 | 0.977317     | 1            |
| 18     | 1238.509            | 4.5          | 1048.469     | 310.7763 | 0.973568     | 1            |
| 19     | 1240                | 4.476397     | 1052.481     | 310.5041 | 0.96926      | 1            |
| 20     | 1240                | 4.497986     | 1068.305     | 311.8831 | 0.962031     | 1            |
3.3 Microstructure for Astm A995/A995M

A metallurgical microscope is used to study the changes in the morphology of the dendritic structure of the annealing in ASTM A995/A995M Duplex Stainless steel under the selected process parameters. Microstructures of 100X magnification were shown in Figure 5. With the increment of Heating temperature, a significant elimination in micro-porosity and other defects were noted through the quantitative metallographic analysis as discussed earlier. The casted ASTM A995/A995M microstructure region shows two different colors with white and black. The white region is Austenite and a black area is Ferrite, thus the ferrite changes into needle like structure while the temperature increases which results in the increased hardness. The dendritic branches act as the load carrying member. Mechanical properties of the alloy were increased by the refinement of dendritic branches. The alloy samples of ASTM A995/A995M at a heating temperature of 1240°C with a holding time of 4.5 hrs and a cooling temperature of 1043.9080°C has the increased austenite content which results in improved hardness.
3.4 Validation

The validation was done to observe the deviations of experimental values and predicted values. The deviation between the experimental and predicted value gives a details of residuals (e). The Figure 6 shows the deviation of experimental values and predicted values; higher the deviations of data points from mean line higher the residuals. The figure shows the data points closer to the mean line; thus the residuals become lesser ad increases the accuracy of prediction. The Table 2 shows the deviation in prediction of hardness for desirability analysis results; it is observed from the table, the ‘e’ falls less than 2 for hardness thus the observation of lower residuals reveals that the predicted value is highly acceptable and accurate.

| Sl no | Injection pressure (A) | Shot Velocity (B) | Furnace Temperature (C) | Brinell hardness (MH) (Predicted) | Brinell hardness (MH) (Experimental) | Residual (MH) |
|-------|------------------------|-------------------|-------------------------|----------------------------------|--------------------------------------|--------------|
| 1.    | 208.981                 | 0.6               | 677.83                  | 125.48                           | 124.23                               | 1.25        |

4. Conclusion

Thus from the above chapters we can able to conclude that the austenite percentage in the duplex stainless steel is the only factor which determines the hardness of the material. Since this project is to improve hardness of the material, so the increase of austenite content in the material will automatically increases the hardness. Thus from this research work, on conducting DOE and various experiments regarding hardness, the heating temperature of 1240°C with holding time of 4.5hrs and cooled up to 970°C provides the maximum hardness of 311 without changing the material. So we suggest these parameters to the valve manufacturing industries to improve hardness of their product.
5. References

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