Thesis for the Degree of Master of Engineering

Weibull Statistical Properties for Rockwell Hardness of Incoloy 825 at Different Heat Treatment Temperatures

by

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Department of Materials Science and Engineering

Graduate School

Pukyong National University

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(열처리 온도가 다른 Incoloy 825의 로크웰 경도의 와이블 통계 특성)

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by

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Abstract

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This study was carried out to investigate the microstructure and Rockwell hardness of heat treated 90% hot forged Incoloy 825 alloy. With increasing solution treatment temperature, grain size increased and precipitates decreased, and precipitates not observed at 1,273K. With increasing aging time at 973K, precipitate increased and precipitates size increased. Shape parameter of solution treated material increased with increasing temperature, but decreased sharply at 1,273K. Scale parameter also decreased. Shape parameters and scale parameters of the aging treatment material increased, according to increasing of aging time.

Keyword: Incoloy 825, Rockwell Hardness, Heat Treatment, Weibull Statistics

1. Introduction

Incoloy 825 alloy, based on Ni-Fe-Cr and contains alloying elements such as Co, Ti and Al, is strong against general corrosion, crevice corrosion, grain boundary corrosion, and stress corrosion. In particular, it has excellent corrosion resistance to sulfuric acid, phosphoric acid, and fuel gas containing sulfur etc. So, it is widely used in offshore plants, chemical, and refining industries. It also has excellent high and low temperature characteristics, and is widely used for parts such as nuclear power plants and LNG vessels.

Recently, refining and offshore plant industries using super-heat-resistant alloys such as Incoloy alloys, have been larging their facilities as a way of increasing efficiency of use of the devices. In accordance with these changes, parts manufacturing methods by forging are gradually changing from die forging for manufacturing small parts, to open die forging for manufacturing large parts. However, the large size of parts increases the mass effect, and lowers the forging temperature. As a result, precipitation behavior of precipitates changes. Additionally, Park et al [1] studied grain size depending on the forging ratio. So, it is necessary to develop an optimal process for hot forging and heat treatment, to secure stability and expand the range of use of these alloys.

Incoloy 825 alloys are increasingly used in a various industries because of good characteristics. Raymond [2] studied on mechanisms of sensitization and stablization of Incoloy 825 in the 922-1,033K range. Hussain et al [3] studied high-temperature oxidation and spalling behavior of heat-treated Incoloy 825 during 2 min - 100 h in the temperature range from 873K to1573K. Shaikh et al [4] were solution treated at 1473K for 1h using Incoloy 825. These specimens were then aged at 1,143K for various intervals in the range 1-264h. They studied precipitation of Incoloy 825 by scanning electron microscopy. Dunn et al [5] conducted on prediction research of localized corrosion of Alloy 825 in nuclear waste environments. Pan et al [6] were evaluated sensitization by using corrosion experiment that was correlated to chemistry analyses of grain boundary. Sensitized microstructures were known to include $M_{23}C_6$-shape carbide and chromium deficiency region near the grain boundary. Park et al [1] studied the mechanical properties and microstructure of heat treated 90% hot forged Incoloy 825 alloy.

These characteristics are expected to be greatly influenced by the microstructure, precipitation behavior of precipitates, and grain size etc. So, it is need to investigate the microstructure by heat treatment after the forging process, and to investigate the influence of such microstructural changes on mechanical properties. In particular, mechanical properties are not of definite value, but variable values, so

Weibull [7] mentioned the statistic application about a various problems. Kim et al [8] carried out study about the mechanical properties of ZrO_2 composites by Weibull statistical analysis processing. Ahn et al [9,10] evaluated on Weibull statistical analysis about Vickers hardness of shot-peened ZrO_2 Composites Ceramics and corroded Al_2O_3 Ceramics. Also, Lee et al [11] discussed Weibull distribution about Vickers hardness of Al_2O_3/SiC composite depending on the SiC concentration. Park et al [12] studied about Weibull analysis to shear strength of resistance welded ball studs.

In this study, the Incoloy 825 alloy was hot-forged 90% by open die forging, and the Rockwell hardness was measured by change of solution treatment temperature and aging time. The Rockwell hardness was analyzed by the Weibull statistics.

2. Material and experiment method

2.1. Material

Materials were manufactured by Daido Steel of Japan, using Vacuum Induction Melting (VIM) and Electro Slag Remelting (ESR). Materials were based on the paper of Park et al [1]. Table 1 shows chemical compositions of material.

Table 1. Chemical composition of specimen (wt.%).

C	Si	Mn	S	Ni	Cr	Cu	Mo	Al	Ti	Fe
0.03	0.30	0.60	0.01	45.07	22.2	1.70	3.00	0.10	0.10	26.9

2.2 Hot forging

The hot forging process was generally heated to 1,423 K, to make grain size smaller than required grain size of 50 μm at the Incoloy 825 alloy.[5] Thereafter, after a certain time, a forging ratio of 90 was obtained with a 5,000 ton press. At this time, the finish forging temperature was 1,173K. To investigate the effect of solution treatment temperature, material was maintained at 973K, 1073K, 1173K and 1273K for one hour, and then water-cooled. To investigate the effect of aging time, materials was water cooled after holding of 1h, 5 h, 10 h, and 30 h at 973K.

2.3 Hardness test

Hardness was measured using a Rockwell hardness tester. Load was 980N, and 20 data were measured for each specimen, and used for analysis.

3. Results and Discussions

3.1 Structures observation

Fig. 1 shows optical microscope and scanning electron microscope photographs of Incoloy 825 alloy from result of Park et al [1] In the photograph, twin crystals partially exist, and grain size is austenite single phase structure of 200 ㎛ or more.

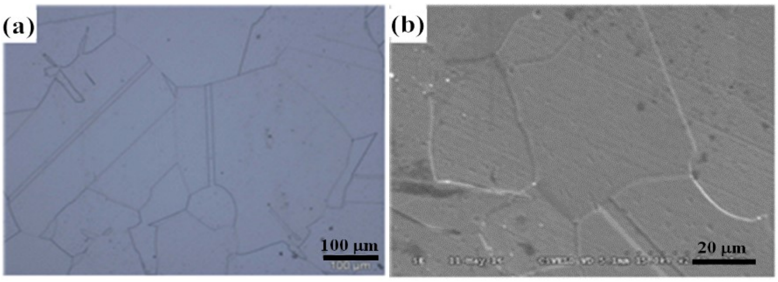


Fig. 1. Micrographs of Incoloy 825. (a) Optical, (b) SEM

Fig. 2 shows the microstructure of the SEM, of the solution-treated specimen. From 973K to 1,173K, it can be seen that precipitates are present partially in the transgranular, as well as the grain boundary of the austenite, regardless of solution treatment temperature. Conversely, solution treated specimen at 1,273K have no precipitates and was an austenite single phase. It is judged that this is a region wherein austenite and carbide coexist, at a temperature of less than 1,223K, and that 1,273K is austenite single phase region. Results are a good match with other research of precipitates in Superalloys. It can be seen that as solution treatment temperature increases, grain size increases. Particularly, crystal grains were much coarsened at 1,273K. This is because the higher solution treatment temperature, the faster the growth of grains occurs. The reason for coarsening at 1,273K is that growth of the austenite grains occurs rapidly without disturbance by precipitates, because it is single phase of austenite.

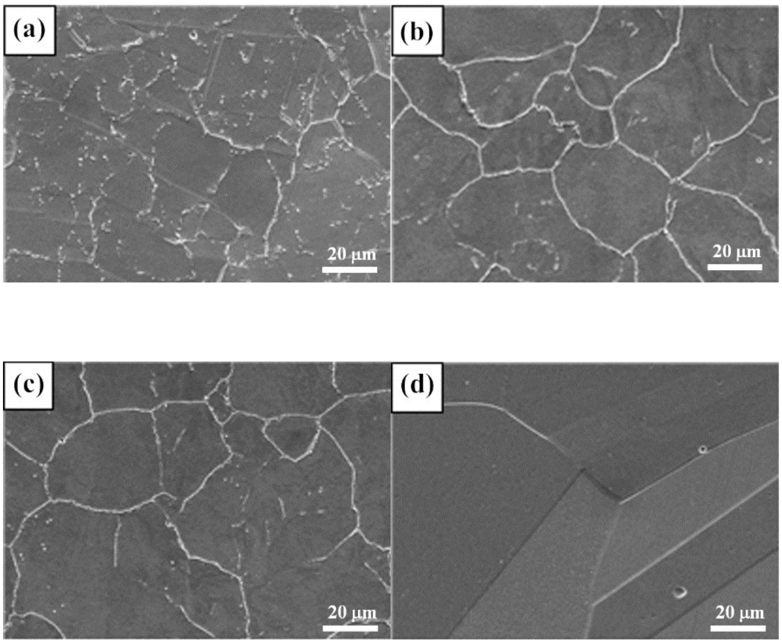


Fig. 2. SEM micrographs in hot forged Incoloy 825 of 90%. (a) 973K, (b) 1,073K, (c) 1,173K, (d) 1,273K

Fig. 3 shows the SEM microstructure to investigate the precipitation depending on aging time. The precipitate was principally precipitated in grain boundaries of the austenite regardless of aging time. Additionally, as aging time becomes lengthier, precipitates are precipitated even in the transgranular, and amount of precipitation increases. However, even when aging time became lengthy, growth of grain did not occur. This is because growth of crystal grains is disturbed by precipitates.

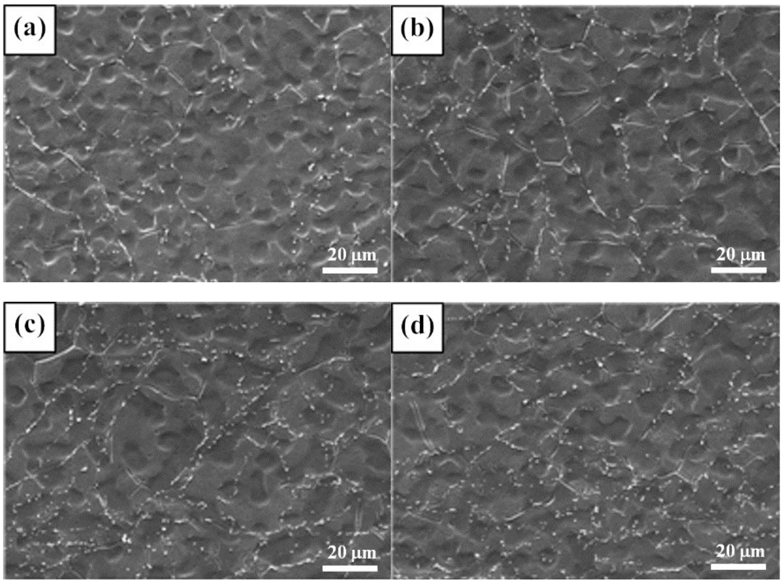


Fig. 3. SEM micrographs according to aging time at 973K in hot forged Incoloy 825 of 90%. (a) 1 h, (b) 5 h, (c) 10 h, (d) 30 h

3.2 Weibull statistical analysis of hardness

Fig. 4 shows the Rockwell hardness of solution treated specimen at 973K, 1,073K, 1,173K and 1,273K for 1 h. The Rockwell hardness decreases as solution treatment temperature increases. 1,073K and 1,173K decreased by approximately 8% compared to 973K, respectively. But 1,273K decreased approximately 12%.

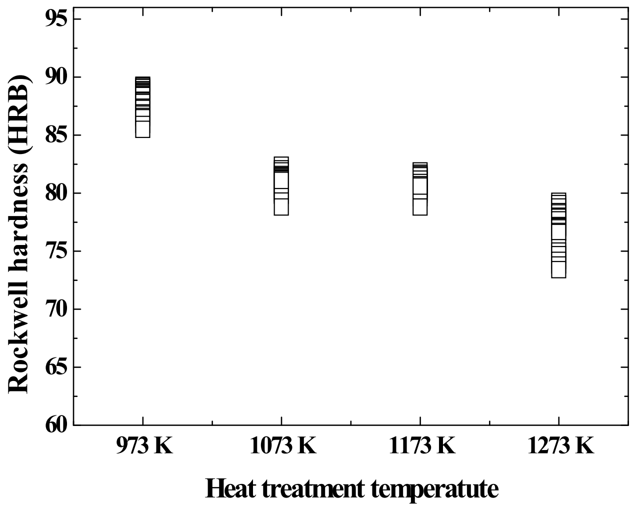


Fig. 4. Comparison of the Rockwell hardness, as function of different solution treatment temperature.

Fig. 5 shows the Rockwell hardness for specimens with aging for 1, 5, 10, and 30 hours at 973K. The Rockwell hardness increased, with increasing aging time. The aging time of 10 and 30 hours compared with 1 hour, increased by 1.8%, 2.1% and 3.7%, respectively.

As shown in Fig. 4 and Fig. 5, hardness is not a definite value, but stochastic quantity that shows variability. The Weibull plot, that is, linear regression method, is the most common method. Cumulative distribution function that gives probability of *P* at the Rockwell hardness, can be expressed by the following equation (1):

$$P = 1 - \exp\left[-\left(\frac{x}{\beta}\right)^{\alpha}\right] \qquad (1)$$

Where *P* is the probability of the Rockwell hardness, and α and β are the shape parameter and scale parameter, respectively. *x* represents the Rockwell hardness. The scale parameter β represents Rockwell hardness at 63.2%, and α is a shape parameter representing data scattering. High α values indicates a small dispersion of property values and uniformity of material.

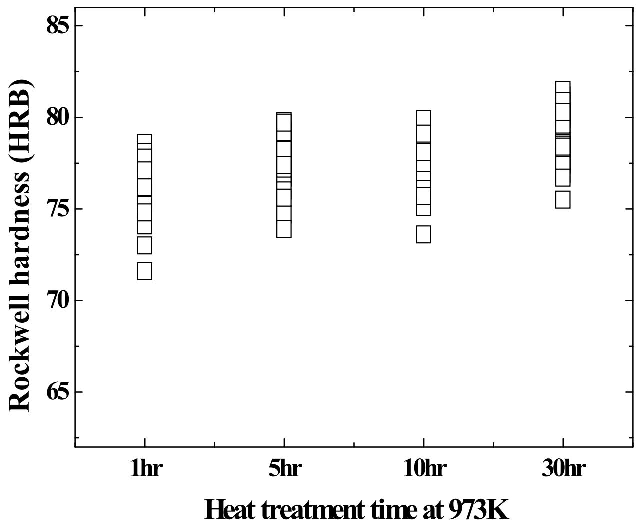


Fig. 5. Comparison of the Rockwell hardness, as function of different aging treatment time at same temperature.

Fig. 6 shows the Weibull probability for the Rockwell hardness of the solution treated specimen at 973K, 1,073K, 1,173K and 1,273K for 1 h. Rockwell hardness was expressed as a straight line, and it can apply to the Weibull probability distribution. Table 2 shows the two parameters of equation (1), estimated from the Rockwell hardness. The table also shows mean, standard deviation (Std), and coefficient of variation (COV) depending on Weibull analysis. From the above results, as solution treatment temperature increases, scale parameter becomes smaller. Shape parameters increased at temperatures between 973K and 1,173K, but decreased sharply at 1,273K. This is judged that the influence of the precipitate and the structure, as explained in Fig. 2.

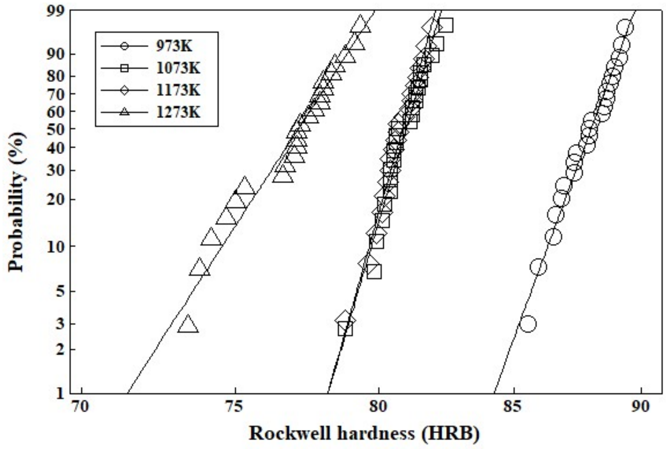


Fig. 6. Weibull plot of the Rockwell hardness, as function of different temperature.

Table 2. Statistical analysis of the Rockwell hardness (B scale) according to different temperatures.

	HRB			Weibull parameters		R^2
	Std.	Mean	COV	Shape parameter	scale parameter	
973K	1.079	87.8	0.012	96.8	88.3	0.99
1,073K	0.808	80.9	0.010	121.0	81.2	0.98
1,173K	0.752	80.7	0.009	127.0	81.1	0.99
1,273K	1.673	76.9	0.022	55.1	77.7	0.98

Fig. 7 shows the Weibull analysis for the Rockwell hardness of the aging treated specimen for 1, 5, 10 and 30h at 973K. Table 3 shows the two parameters of equation (1), estimated from the Rockwell hardness of aged specimen. The table also shows mean, standard deviation (Std), and coefficient of variation (COV) depending on Weibull analysis. From the above results, as aging time increased, the scale parameter and shape parameter increased. This is because the amount of precipitate increased, as explained in Fig. 3.

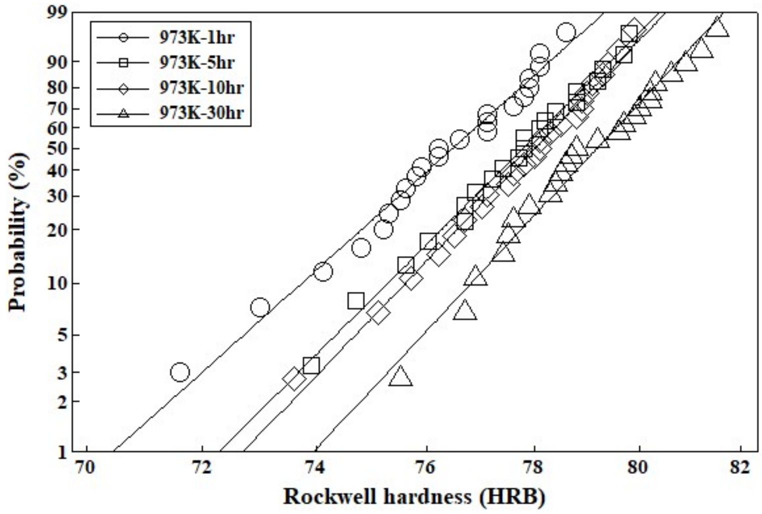
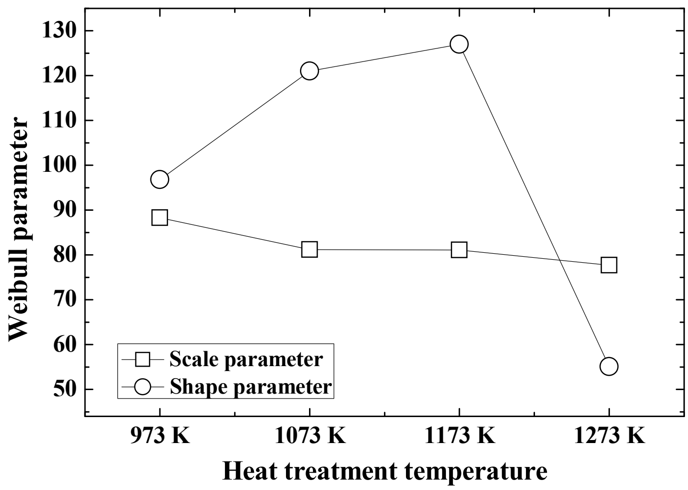


Fig. 7. Weibull plot of the Rockwell hardness, as function of different time at same temperature.

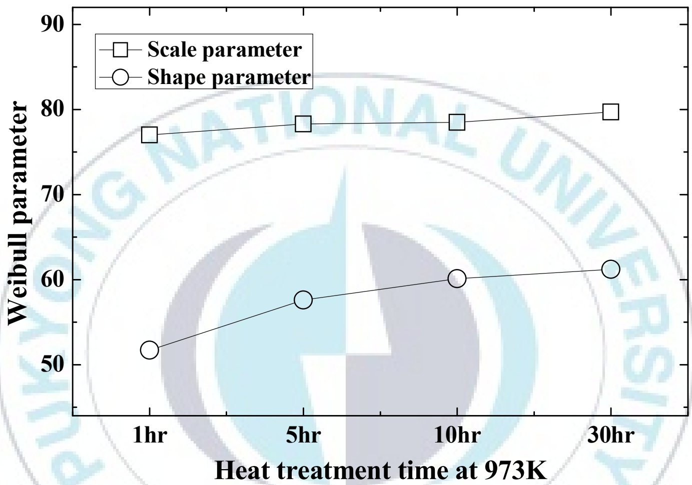
Table 3. Statistical analysis of the Rockwell hardness (B scale) according to different time at same temperature.

	HRB			Weibull parameters		R^2
	Std.	Mean	COV	Shape parameter	scale parameter	
973K-1h	1.739	76.2	0.023	51.7	77.0	0.991
973K-5h	1.578	77.6	0.020	57.6	78.3	0.997
973K-10h	1.528	77.8	0.020	60.1	78.5	0.996
973K-30h	1.525	79.0	0.019	61.2	79.7	0.990

Fig. 8 shows the two parameters obtained from the Weibull analysis of the Rockwell hardness. (a) is a solution treated specimen at 973K, 1,073K, 1,173K and 1,273K for 1 hour, and (b) is an aged specimen at 973K for 1, 5, 10, and 30 h. (a) Shape parameter increased according to increasing temperature, but decreased abruptly at 1,273K. But the scale parameter decreased according to temperature. That is, shape parameters of 1,073K and 1,173K were 25% and 31% larger compare to 973K, respectively, but 1,273K was -43% smaller. Conversely, scale parameters were -8% (1,073K), -8.2% (1,173K) and -12% (1,273K) smaller, respectively. (b) Shape parameters and scale parameters increased, according to increasing of aging time. Shape parameters were larger 11.4% (5h), 16.2% (10h) and 18.4% (30h) compared to 1h. Scale parameters were larger of 1.7% (5h), 1.9% (10h) and 3.5% (30h), respectively.



(a)



(b)

Fig. 8. Shape and scale parameters obtained from Weibull probability of the Rockwell hardness. (a) Effect of heat treatment temperature, (b) Effect of heat treatment time at 973K.

4. Conclusions

Incoloy 825 alloy was hot forging of 90% to investigate the effect of heat treatment on microstructure, and the Rockwell hardness. This study examined microstructure and the Rockwell hardness, for solution treated- and aging treated-materials. In particular, the Rockwell hardness was analyzed by Weibull probability. Grain size became large according to increase of solution treatment temperature, and amount of precipitates in intergranular and transgranular decreased. Precipitates were not observed at 1,273K. The amount of precipitate increased with aging time, and the size of precipitate became larger. The lower the solution treatment temperature, the longer the aging time, and the Rockwell hardness increased. Shape parameter of solution treated material increased with increasing temperature, but decreased sharply at 1,273K. The scale parameter decreased. Shape parameters and the scale parameters of the aging treatment material increased, according to increasing of aging time.

References

1. Park, Y.T., Kim, D.H. and Kang, C.Y., 2017. Effect of Solution Heat Treatment on the Mechanical Properties in Incoloy 825 Alloy. J. of the Korean Society for Heat treatment. 30(3), 99-105.
2. Raymond, E.L., 1968. Mechanisms of Sensitization and Stablization of Incoloy Nickel-lron-Chromium Alloy 825. Corrosion. 24(6), 180-188.
3. Hussain, N., Schanz, G., Leistikow, S. and Shahid, K.A., 1989. High-Temperature Oxidation and Spalling Behavior of Incoloy 825. Oxidation of Metals. 32(5–6), 405–431.
4. Shaikh, M.A., Iqbal, M., Ahmad, M., Akhtar, J.I. and Shoaib, K.A., 1992. Precipitation Study of Heat-Treated Incoloy 825 by Scanning Electron Microscopy. Journal of Materials Science Letter. 11(14), 1009-1011.
5. Dunn, D. S., Sridhar, N. and Cragnolino, G. A., 1996, Long-Term Prediction of Localized Corrosion of Alloy 825 in High-Level Nuclear Waste Repository Environments, Corrosion, 52(2), 115-124.
6. Pan, Y. M., Dunn, D. S., Cragnolino, G. A. and Sridhar, M., 2000. Grain-boundary chemistry and intergranular corrosion in alloy 825, Metallurgical and Materials Transactions A. 31, 1163–1173
7. Weibull, W., 1951, A Statistical Distribution Function of Wide Applicability. Journal of Applied Mechanics. 18, 293-305.
8. Kim, S.J., Kim, D.S. and Nam, K.W., 2015. Determining Mechanical

Properties of ZrO_2 Composite Ceramics by Weibull Statistical Analysis. Trans. Korean Soc. Mech. Eng. A. 39(10), 955-962.

9. Ahn, S.H., Kim, D.S. and Nam, K.W., 2016. Weibull Statistical Analysis According to Vickers Indentation Load of Peened ZrO_2 Composites Ceramics by Different Shot Size. Trans. Korean Soc. Mech. Eng. A. 40(12), 987-995.
10. Ahn, S.H. and Nam, K.W., 2017. Temperature-Dependent Characteristics of Weibull Statistical Analysis for Vickers Hardness of Corroded Al_2O_3 Ceramics. Journal of Ceramic Processing Research. 18(9), 646-658.
11. Lee, K.H. and Nam, K.W., 2018. Weibull Statistical Analysis for Vickers Hardness of Al_2O_3/SiC Composite according to the SiC Concentration. Journal of Ceramic Processing Research. 19(1), 75-79.
12. Park, I.D., Nam, K.W. and Kang, C.Y., 2018. Shear Strength of Ball Studs According to the Resistance Welding Conditions & Reliability Testing Based on the Weibull Distribution Function. Journal of Mechanical Science and Technology. 32(12), 5647-5652.

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