

A Statistical Approach to the Heat Treatment Optimization of Al-Al_2O_3 Particulate Composites

*G. Leisk and A. Saigal**

The effects of three heat treatment variables—solutionizing time, aging time, and aging temperature—on the yield and ultimate tensile strength of alumina/aluminum metal matrix composites were studied using a statistical technique known as the Taguchi Method. Composites consisting of 10, 15, and 20 volume percent alumina particulate reinforcements in a 6061 alloy matrix were investigated. Fractional factorial experimentation was performed using three levels for volume percentage of reinforcement and the three heat treatment parameters. Based on the Taguchi analysis, the optimal combination of heat treatment variables for each reinforcement volume percentage of these alumina/aluminum composites consists of 6 hours (instead of the standard 2 hours) of solutionizing at 985 °F (529 °C) followed by aging at 320 °F (160 °C). The predicted optimal strength correlated very closely to actual results obtained during experimentation. SEM micrographs illustrate the microstructural differences that occur as a result of the various combinations of heat treatment.

1 Introduction

COMPOSITE materials are generally classified into one of three categories: polymers, ceramics, and metals. Composites have been at the forefront of materials research since the 1960s because of the interesting property combinations that can be attained and the way they can be tailored to specific applications.[1] Metal matrix composites (MMCs) have grown in popularity in recent years due to the high specific strength and stiffness that can be obtained. Other potential benefits of MMCs include lower coefficient of thermal expansion and greater wear resistance than is presently attainable in conventional alloys.[2]

There are three major classes of metal matrix composites depending on the type of reinforcement in the matrix: fibers, whiskers, and particulates. Particulate metal matrix composites, the focus of this study, tend to be homogeneous and isotropic and can be formed using conventional metal deformation processes.[3] They can, therefore, be both easier to implement because of their conformance to conventional design techniques and more cost efficient than other composites, which need special fabrication and processing techniques.[4]

It has been shown that the presence of reinforcements in a matrix alloy can alter the heat treatment response of a MMC. Therefore, the optimum heat treatment procedure for any given MMC can differ from that of the unreinforced alloy.[5,6,7] In the traditional manner of experimentation (known as full factorial experimentation), tests are run for each combination of variables that is identified. Due to the numerous combinations of material and heat treatment variables that were to be tested for this composite, an extremely large number of experiments would have to be conducted. In order to save both time and resources, statistical methods have been developed which reduce the number of experiments necessary to a practical level (known as partial or fractional factorial experimentation).[8] This paper presents one such statistical analysis technique known as the Taguchi method as applied to the optimization of the heat treatment of alumina/aluminum metal matrix composites.

G. Leisk and **A. Saigal,** Department of Mechanical Engineering, Tufts University, Medford, Massachusetts 02155.

2 Experimental Procedure

The materials investigated in this paper are alumina-reinforced aluminum 6061 alloy matrix composites. The composition of the 6061 alloy is 1.0% Mg, 0.6% Si, 0.28% Cu, and 0.2% Cr. The alumina reinforcement is in the form of particulates approximately 12 to 15 micrometers in diameter. The samples investigated consisted of 10, 15, and 20 vol% alumina reinforcements. The raw material was supplied by Duralcan USA in the form of rectangular bars 36″ × 3″ × 0.75″ (914 mm × 76 mm × 19 mm) in the as-extruded condition. The bars were extruded from 7″ (178 mm) DC cast billets at 800 °F (427 °C) and 20 ft/min (6.1 mm/min) exit speed.

The bars received from Duralcan were machined using solid carbide and diamond tooling on a Bridgeport CNC to obtain standard dog-bone specimens for mechanical testing as per ASTM specification for Tension Testing of Wrought and Cast Aluminum-Alloy Products.[9] The dimensions of the specimens are shown in Fig. 1.

After heat treatment, the tensile specimens were tested in uniaxial tension using a computer-controlled Instron 4505 tensile test machine and a 22,000 lb (100 kN) load cell. The specimens were loaded at the rate of 0.02 in./min (0.51 mm/min). Upon rupture of each specimen, transverse cuts in each specimen were made with a diamond cut-off wheel away from the area of rupture. Each cross-sectional surface was then polished metallographically. A scanning electron microscope (SEM) was used with 1000× magnification to observe the microstructural changes that occurred as a function of the heat treatment combination used.

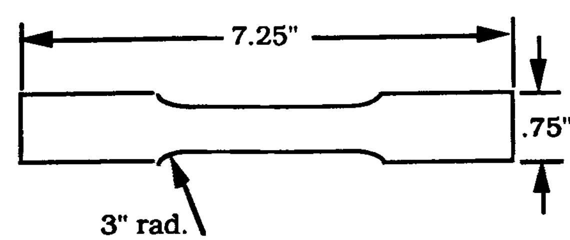

Fig. 1 Tensile test specimen shape and dimensions.

Table 1 Orthogonal Array for Taguchi Analysis

Run	Reinforcement, vol%	Solutionizing time, h	Aging time, h	Aging temperature
1	10	2	12	260 °F (127 °C)
2	10	6	18	320 °F (160 °C)
3	10	12	24	380 °F (193 °C)
4	15	2	18	380 °F (193 °C)
5	15	6	24	260 °F (127 °C)
6	15	12	12	320 °F (160 °C)
7	20	2	24	320 °F (160 °C)
8	20	6	12	380 °F (193 °C)
9	20	12	18	260 °F (127 °C)

3 Taguchi Method

Dr. Genichi Taguchi has two basic beliefs that are fundamental to his statistical method. The first is that a reduction in variation of a product or process represents a lower loss to society. His second belief is that proper development strategies can reduce variation. The purpose of running experiments, therefore, should be to reduce and control the variation in a product or process.[10] The adaptation of this method to materials testing allows for investigation of many more variables than would be practical in a full factorial experiment. For instance, if the goal is to optimize certain material strength characteristics, specific variables that greatly influence the strength can be identified and included in the fractional factorial experiment.

The combination of variables levels that are used in each experimental situation have been predetermined by Taguchi. Each variable and its variable level, as determined by the experimenter, are assembled into a table known as an orthogonal array. Taguchi identifies two classifications of variables (factors) that contribute to product functionality: controllable and uncontrollable (noise) factors. Controllable factors are variables in the production process that can be easily changed. In this experiment, three heat treatment variables and the volume percentage of alumina are introduced as controllable factors. Uncontrollable (noise) factors are variables that are either too difficult or too expensive to be changed. Because noise factors are usually tough to reduce or eliminate entirely, a goal of the Taguchi method is to lessen the impact of the noise factors.[10] For this investigation, noise factors are not included in the experimental setup, as is most often the case. Instead, three replications of each experimental run are performed in order to analyze the noise and to study the variation from the mean response.

4 Taguchi Analysis of Heat Treatment

In the standard T6 heat treatment, specimens are usually solutionized at 985 °F (529 °C) for 2 h, quenched in room-temperature water and, finally, artificially aged at 320 °F (160 °C) for 16 to 20 h. As discussed previously, since the alumina/aluminum composites consist of reinforcement particulates embedded within an alloy matrix, the optimal heat treatment is expected to deviate from the standard T6 heat treatment for alloys. Selection of the specific heat treatment variable levels used are based on several sources. For example, for solutionizing time, the first level is 2h as suggested in the standard T6 heat treatment. The last level, 12 h, is the optimal solutionizing time for two separate particulate SiC reinforced cast composites that Labib *et al.*[11] and Hammond[6] have investigated. The 6h intermediate level is another frequently recommended solutionizing time.

In this study, three levels for each of the three main heat treatment variables were selected: solutionizing times of 2, 6, and 12 h; aging times of 12, 18, and 24 h; and aging temperatures of 260, 320, and 380 °F (127, 160, and 193 °C). Each of these combinations would be applied to the three composites containing 10, 15, and 20 vol% alumina reinforcements. In the traditional method of experimenting, all of these variables would lead to 81 separate heat treatments and tensile tests. However, including three replications of each experimental run, only 27 specimens have to be machined and tested in this fractional factorial experiment.

The orthogonal array with the specific levels of each factor for optimizing the heat treatment of alumina-reinforced aluminum 6061 alloy matrix composites is shown in Table 1.

5 Results and Discussion

Three specimens were machined and then heat treated for each run number in the orthogonal array in Table 1. Each specimen was then tested until fracture on the Instron 4505 tensile test machine. SADIE (Speedy Analysis and Design of Industrial Experiments), an IBM PC-based Taguchi-type software program, was used to help analyze the experimental results.[12]

Software programs like SADIE program averaging algorithms on the orthogonal arrays to analyze factor effects and to predict combinations of variable levels that would produce optimal results using a technique known as ANalysis Of VAriance (ANOVA). In general, ANOVA is a statistical method that determines the relative contributions of the factors by comparing their variances. Optimal performance is then calculated in ANOVA by adjusting the mean response for each experiment by including each individual factor level contribution.[8]

In mathematical terms, the mean response of an experiment is given by:

$$\bar{y}_i = \sum_{j=1}^{J} \sum_{k=1}^{K} {y_{ijk}}/{JK}$$

where y_{ijk} = response for i*th* control and j*th* noise row, and k*th* replication; J = number of noise rows; and K = number of replications. Using this equation, the same amount of information that would be achieved by running a full factorial set of experiments can be obtained. In addition to the mean response calculations, the variation in the mean response based on the number of replications performed is calculated by using the standard deviation formula:[13]

$$\text{Variation} = \sqrt{\sum_{j=1}^{J} \sum_{k=1}^{K} {\left(y_{ijk} - \bar{y}_i\right)^2}/{(JK - 1)}}$$

The variation of the responses about the mean value due to experimental noise is also important. A signal-to-noise (S/N)

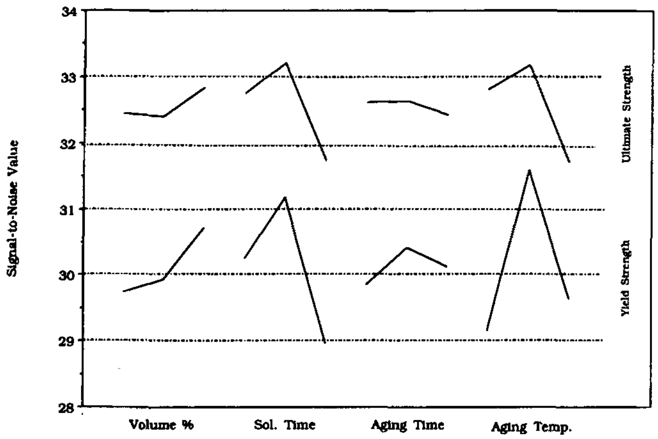

Fig. 2 Signal-to-noise value factor effects.

value is introduced to provide a method for numerically comparing factors and factor levels for both mean response and variation. Taguchi describes any product or process response that does not have a negative value and for which the best value would be infinity as a 'bigger is better' response. In the case of alumina/Al MMCs, the goal is to achieve the highest yield and ultimate strength values possible so that a 'bigger is better' expression for the signal-to-noise is used:[14]

$$\frac{S}{N} = -10 \cdot \log_{10}\left(\frac{1}{n}\left(\Sigma \frac{1}{y_i^2}\right)\right)$$

where n = number of data points, and y_i = mean response for each run.

Figure 2 shows the effects various factors have on the yield and ultimate tensile strength, based on the S/N ratio response, of the alumina/Al MMCs. The factors with the biggest effect appear to be aging temperature on the yield strength and solutionizing time on the ultimate strength. Both the yield and ultimate strength responses are relatively insensitive to the aging time to which the composite is subjected to. In addition to the factor effects, Fig. 2 also indicates that the middle levels of the solutionizing time, aging time, and aging temperature result in the highest yield strength and ultimate tensile strength.[15]

By examining the predicted mean response data statistically for each individual volume percentage of alumina using SADIE, the optimal heat treatment parameter combination can be predicted. Since variation from target response is a concern, experimental responses that are used in this optimization are analyzed in a S/N format as discussed above. For alumina/Al MMCs, the optimal heat treatment combination predicted consists of 6 h of solutionizing at 985 °F (529 °C) followed by 18 h of aging at 320 °F (160 °C). As can be seen from Table 1, this particular combination was tested for the 10% alumina reinforcement composite. Since the predicted results are based on the experimental runs, yield strength and ultimate tensile strength for the 10% alumina-reinforced composite samples that have been optimally heat treated are illustrated by the three test replications of experimental run 2. These results are presented in Table 2.

Table 2 Test Results for 10% Alumina Composite After Optimal Heat Treatment

	Replication 1	2	3	Mean	Std. dev.
Yield strength, ksi	42.68	42.54	42.14	42.45	0.28
Ultimate tensile strength, ksi	48.70	49.12	48.57	48.80	0.29

Table 3 Mean and Deviation Yield Strength Responses for 6-h Solutionizing Time and 320 °F Aging Temperature

Factor/level Vol%	Age time, h	Mean response, ksi	Standard deviation
10	12	40.38	0.79
	18	42.45	0.28
	24	41.39	0.10
20	12	43.99	1.57
	18	46.06	0.56
	24	45.00	0.21

Table 4 Mean and Deviation Ultimate Strength Responses for 6-h Solutionizing Time and 320 °F Aging Temperature

Factor/level Vol%	Age time, h	Mean response, ksi	Standard deviation
10	12	48.71	0.57
	18	48.80	0.29
	24	48.02	0.10
20	12	50.51	2.01
	18	50.60	1.01
	24	49.82	0.35

Since the variability of the responses due to individual factors is so important, both the mean responses and the standard deviations are presented in Tables 3 and 4. Tables 3 and 4 show these values for yield strength and ultimate tensile strength as a function of aging time for the optimal solutionizing time (6 h) and aging temperature (320 °F/160 °C) for both the 10% and 20% alumina/Al composites, respectively. Although it appears that the variability of the mean responses for both yield and ultimate strength is lower for 10% alumina composites as compared to 20% alumina composites, and can be reduced by using longer aging times, the overall insignificance of the values shown indicate that any aging time chosen produces acceptable amounts of response variability.

SEM micrographs are shown in Fig. 3 and 4. Figure 3 shows a typical microstructure of an alumina/Al composite containing 10 volume percentage reinforcement particulates subjected to heat treatment under the conditions of experimental run 1. Figure 4 shows the microstructure of a similar 10% composite upon heat treatment under the conditions of experimental run 2, which is predicted to be the optimal heat treatment combination.

6 Conclusions

From the experimental method used and the results obtained, the following conclusions can be drawn. Fractional factorial ex-

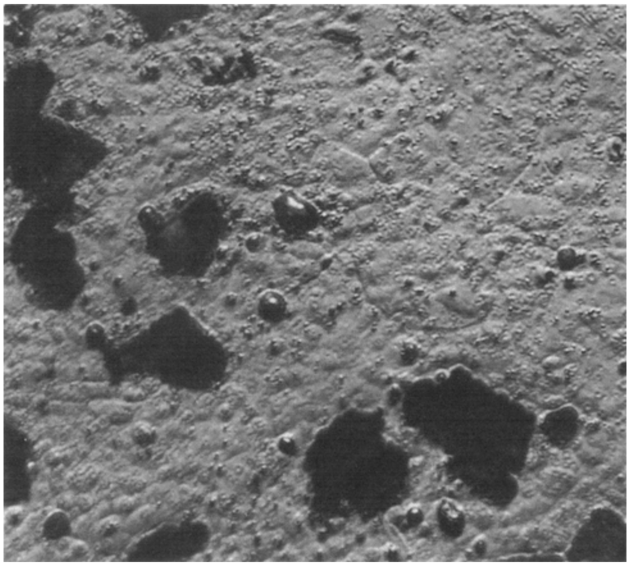

Fig. 3 Microstructure of 10% composite after experimental run 1 heat treatment. 1000×

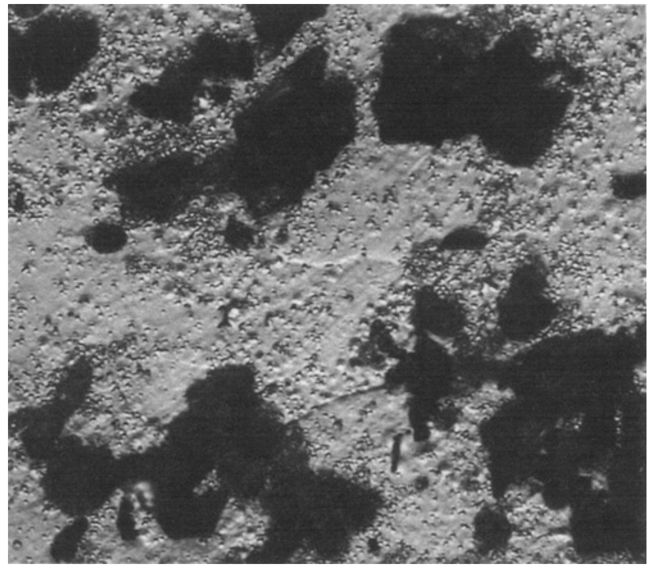

Fig. 4 Microstructure of 10% composite after experimental run 2 (optimal) heat treatment. 1000×

perimentation using the Taguchi method can be effectively applied to materials testing. Signal-to-noise ratio factor effect plots indicate that solutionizing time and aging temperature significantly affect both yield and ultimate tensile strengths of alumina/Al MMCs. Aging time, however, can probably be set at any level within the limits shown here with minimal effect on the ultimate tensile strength of the composite. In addition, it is recommended that the alumina-reinforced aluminum 6061 alloy composites be solutionized for 6 h instead of the standard 2 h for higher strength. SEM observations show typical effects that the various heat treatment combinations have on 10% alumina particulate/aluminum 6061 metal matrix composites.

Acknowledgment

The authors would like to thank Mr. Michael D. Skibo of Duralcan USA for providing the bar stock for this study.

References

1. M.A. El Baradie, *J. Mater. Processing Technology*, *24*, 261 (1990).
2. A. Saigal, *Proceedings of Symposium on Damage and Oxidation Protection in High Temperature Composites*, *2*, 89 (1991).
3. M. Manoharan and J.J. Lewandowski, *Scr. Metall.*, *23*, 1801 (1989).
4. S.V. Kamat, J.P. Hirth, and R. Mehrabian, *Acta Metall.*, *37*, 2395 (1989).
5. S. Dionne, M.R. Krishnadev, and R. Bouchard, *Metal & Ceramic Matrix Composites: Processing, Modeling & Mechanical Behavior*, 243 (1990).
6. D.E. Hammond, *AFS Trans.*, 587 (1989).
7. I. Dutta, S.M. Allen, and J.L. Hafley, *Metall. Trans.*, *22A*, 2553 (1991).
8. R.K. Roy, *A Primer on the Taguchi Method*, Van Nostrand Reinhold, New York, 41 (1990).
9. ASTM Standard Designation B 557-84, 49 (1989).
10. P. Ross, *Taguchi Techniques for Quality Engineering*, McGraw-Hill, New York, 1-169 (1988).
11. A. Labib, H. Liu, A.S. Rezk, and F.H. Samuel, *AFS Trans.* (1992).
12. R.F. Culp, *Speedy Analysis and Design of Industrial Experiments (SADIE) Manual* (1988).
13. *AT&T, AT&T Quality Workbench ROBUST User Manual*, D-1 (1989).
14. G. Taguchi, *Introduction to Quality Engineering*, Asian Productivity Organization, New York, 77-135 (1986).
15. A. Saigal and G. Leisk, *Scr. Metall.*, *6* (1992).