



Development of Rapid Tooling Material Formula Based on Aluminum Filled Epoxy Resin†

GUANG YANG^{1,2,*}, SHI-XIONG MA¹, YAN-FANG YUE¹, YONG-DI ZHANG¹ and LIN-NAN HE¹

¹School of Mechanical Engineering, Hebei University of Science & Technology, Shijiazhuang 050018, P.R. China

²State Key Laboratory for Manufacturing Systems Engineering (Xi'an Jiaotong University), P.R. China

*Corresponding author: Tel: +86 13831169836; E-mail: y_guang@126.com

AJC-15714

This paper examined a new pouring process that utilized an aluminum filled epoxy resin rapid tooling material. The pouring parameters were optimized through variances in the amount of curing agent, diluent and filler. The study comprised of four factors and three orthogonal levels. The optimal formula was determined through range analysis, fracture morphology through scanning electron microscopy (SEM) and analysis of the material combination between the internal interfaces. Aluminum filled epoxy resin materials presented optimized mechanical performance for use as mold materials.

Keywords: Composite material, Orthogonal experiment, Performance analysis, Scanning electron microscopy.

INTRODUCTION

The mechanical and electrical insulation property of epoxy resin and its adhesive performance with various materials make it for other thermosetting plastic. Thus it can be use into coating, composite materials, casting materials, adhesives, moulding materials and injection molding materials and its major end markets include automotive, construction, textile, aerospace industries, etc. Quan and Zhang¹ developed a method to fabricate superhydrophobic surfaces with epoxy resin microspheres. Song *et al.*² used a facile approach to modify the surface of natural cotton fibers with sulfonate groups through epoxy reaction. Zhang *et al.*³ proposed an optimum method to prepare thin foil transmission electron microscopy (TEM) lamellae of multiphase porous functional ceramics *i.e.*, prefilling the pore space of these materials with an epoxy resin prior to focused ion beam milling. Through this, epoxy is to help to maintain the structural integrity of the entire lamella.

Epoxy resin base materials are also widely applied, so in this article we will introduce the application of aluminum filled epoxy resin material in rapid tooling.

Rapid tooling production represents a new milestone for the production of medium and small batch products that can be introduced quickly in a short time to meet greater user demand. Rapid tooling manufacturing thus greatly reduces cost and investment risk in new product development, shortens the cycle of development and launch⁴, provides small batch deve-

lopment momentum and many varieties with the ability for quick modification. Resin-based rapid tooling composite material is malleable under standard temperature and pressure conditions for adaptable use in cavity pouring. It is advantageous in providing short curing time, smooth chemical reaction, small volume shrinkage and minimal shrinkage and residual stress while curing⁵. The material has the advantages of high shape stability, rigidity, impact toughness, hardness and processing performance which not only shortens mold production time and reduces production costs⁶, but also improves mold machining precision. The rapid development of resin-based rapid tooling has led to many advancements in the mold industry.

The research in this area has focused on the influence of isolated factors on the material, while there have been few reports on the curing process and formula. Herein, the mechanical properties of aluminum filled epoxy resin materials were optimized to achieve performance as a mold material.

Aluminum filled epoxy resin mold material components: Aluminum filled epoxy resin (CAFÉ) mold utilizes a rapid prototyping master pattern for the pouring of aluminum matrix composites at room temperature to produce a mold filled epoxy resin⁷. Epoxy resin mold composite material is a high performance mold resin achieved through a curing reaction, which consists of a matrix resin, curing agent and filler and fiber reinforced materials. The components of this are described as follows:

†Presented at 2014 Global Conference on Polymer and Composite Materials (PCM2014) held on 27-29 May 2014, Ningbo, P.R. China

Choice of epoxy resin: After an epoxy resin has been cured, the resulting product demonstrates better cohesion and more compact molecular structure. As a result, it has enhanced mechanical performance and thus is more efficient for mechanical machining. The shrinkage rate in curing is small, which guarantees consistency in product size and small, stable amounts of internal stress. The low molecular weight of epoxy resin provides good liquidity at room temperature, mixed reactions with a variety of curing agents and fillers, convenient operation, enhanced process performance and achievable curing within 0-180 °C. It was thus determined that epoxy resin was the most suitable for the development of rapid tooling and the general epoxy resins E44 and E51 were used as matrix materials in this test for contrast experiments⁸.

Curing agent: The selection of an efficient curing agent is very important. Epoxy resin can present itself in liquid, viscous form and solid states. It has a small single value and can only be produced with a curing agent through a closed loop, condensation, catalysis or addition reaction. An insoluble and infusible three-dimensional mesh structure is then formed and the epoxy resin becomes a valuable polymer. Polyamide resin 650 molecules consist of a fat long carbon chain and polarity amide groups, which allows curing in epoxy resin at both room temperature and under heating conditions. As such, the molecules have a wide scope for use. In addition, they provide a strong sticky relay, toughness, good insulation performance, water resistance, abrasion resistance, impact resistance and good thermal shock performance for use in cured epoxy resin.

Methyl tetrahydrophthalic anhydride has all of the typical features of an anhydride curing agent. An epoxy resin cured with methyl tetrahydrophthalic anhydride has many advantages, such as electrical insulating properties, good mechanical strength and heat resistance. With good comprehensive performance and relatively low costs, it has been widely used in epoxy resin curing. On the other hand, benzene dimethyl amine has good aliphatic amine reactivity and shares a variety of characteristics with aromatic amine. With curing achievable at room temperature, non-toxic smoke and simple operation, it has been widely utilized in aerospace, military industry, machinery, mold, coatings and various anticorrosion materials. It has previously been used as part of the synthesis of several ionic additives and a synthesis of functional epoxy resin. Due to their superior properties, the three aforementioned curing agents were selected for contrast tests in the experiment.

Diluent: Epoxy diluent is important in reducing the viscosity of curing systems, increasing liquidity, prolonging service life when mixed with a basic resin and improving operational functions, while leaving the essential properties of the cured material unaffected. It has convenient application during casting, perfusion, bonding, sealing and dipping. Epoxy diluents are divided into two types *i.e.*, active diluents and nonactive diluents. Active diluents can dilute resins, participate in cross-linking curing reactions and contain active epoxy group in their structure. On the other hand, nonactive diluents cannot participate in the curing reaction and are only capable of diluting the epoxy resin. In this experiment, the active diluents butyl glycidyl ether (BGE) and nonactive diluent dibutyl phthalic acid were selected for the contrast test.

Fillers: As a component of epoxy resin mold, filler (such as aluminum powder) has an important influence on the mechanical performance of mold. The rational use of filler can improve the performance of epoxy resin mold material by reducing shrinkage and the thermal expansion coefficient, improving thermal conductivity and mechanical strength. In addition, filler is relatively inexpensive and can thus contribute to a reduction in the manufacturing costs of epoxy resin mold. Aluminum powder is also effective in improving thermal conductivity, heat resistance, wear resistance, surface precision, bond strength and bending strength. Three measures were chosen for the contrast test: 200, 600 and 800 mesh. Quartz powder is known to increase material tensile strength, bending strength, hardness, insulation performance and reduce shrinkage. It was thus determined that the 500 mesh condition of quartz powder would be used. Graphite powder can improve thermal conductivity, wear resistance and colour in materials. As a result, the 500 condition of graphite mesh was chosen. Aluminum hydroxide powder and ferroferric oxide powder are also known to improve tensile strength, bond strength and the hardness of mold material. Therefore, 600 mesh aluminum hydroxide powder and ferroferric oxide powder and aluminum powder were chosen for the contrast test.

Strengthening material: Short glass fiber (length of 1.5 mm non-alkali chopped glass fiber) is known as an efficient strengthening material for improved bending strength. However, too much length can be detrimental to the material fluidity, while too much shortness is detrimental to the desired toughening effects. In consequence, 1.5 mm short glass fiber (SGF) slices were chosen.

Antifoaming agent: Antifoaming agent is beneficial in preventing the formation of bubbles during the epoxy resin and hardener reaction, reducing residual bubble build-up on the resin curing surface, enhancing the mechanical properties of the cured product and improving the quality of the cured surface. Antifoaming agent dissolves in foam liquid and works to reduce surface tension in bubbles. Partial reduction in the surface tension of the bubble and around the bubble produces little change. However, strong adhesion to a lessened area of surface tension allows extension and finally leads the bubbles to burst. The antifoaming agent dimethyl silicone has low surface energy and surface tension, which provides low solubility and high activity in water and oil. The main chain is composed fundamentally of a silicon-oxygen bond as opposed to a non-polar molecule. It possesses no affinity for polar solvent water and little affinity for general oil; it has chemical inertness, low volatility, high stability and low toxicity. As a result, dimethyl silicone was identified as the optimum antifoaming agent for the experiment.

EXPERIMENTAL

Pouring process design

Test platform: For the pouring of the mold material, a vacuum injection machine ZK-800 was obtained from Shaanxi Hengtong Intelligent Machine Co. Ltd. An XMTD digital display control instrument was used in the vacuum mixing chamber to control temperature. A heat lamp was utilized to provide a consistent resin temperature and a thermocouple

inducted resin temperature (Fig. 1). Moreover, an electro-thermal blowing drying oven was obtained from 101-4, Beijing Ke Wei Yong Xing Instrument Co. Ltd. and used to facilitate constant temperature during poured bar curing. A micro-computer control electronic universal testing machine was obtained from CMT7104 Shenzhen Xin San Si Measurement Technology Co. Ltd. and used to measure material tensile strength and bending strength. A 30 tons hydraulic universal testing machine of material WE-30 was obtained from the Guangzhou Factory of Testing Materials for the measurement of compressive strength. In addition, a vertical milling machine X53K was obtained from Beijing First Machine Tool Plant for milling the end face of the test piece. A brinell hardness tester was also obtained from Shandong Lai Hua Machinery Equipment Factory for material hardness measurement. A C6140 lathe was obtained from Beijing First Machine Tool Plant in order to turn test pieces, while a Hitachi S-4800 scanning electron microscope (SEM) was obtained for the observation of material fracture morphology. Finally, a metallographic microscope was obtained to observe the consistency of the mixed material.

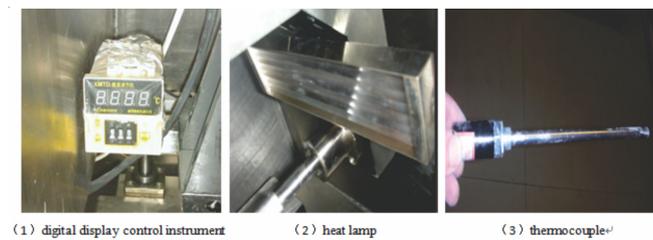


Fig. 1. Improvement of vacuum injection machine

Study of pouring process parameters

Mixing time: Insufficient mixing time can lead to a lack of hardening and inconsistent mixing. The bubble is subsequently unable to discharge in time, leading to easy detachment of the filler and a gathering phenomenon. An excessive timespan leads to unrestrained pouring due to an increase in viscosity. In this study, the agitation speed of the vacuum injection machine was set at 90 r/min and the materials were stirred at intervals of 10, 17, 25 and 32 min. The metallographic experiment was then performed for the first three sample groups, as shown in Fig. 2. The low multiples of the metallographic microscope produced highly efficient uniform mixing. As shown in Fig. 2, a bright spot could be clearly distinguished, which was identified as the metal aluminum powder. The remaining black part was identified as the material base. This inconsistent mixture indicated insufficient mixing time. At 25 min, the material was sufficiently mixed. However, by 32 min, the material had become sticky with poor liquidity; it thus demonstrated difficulties in pouring. As a result, 25 min was chosen as the optimum material mixing time.

Number	E51 (g)	600 mesh aluminum powder (g)	Benzene dimethyl amine (g)	BGE (g)	Quartz powder (g)	1.5 mm SGF (g)	Dimethyl silicone (g)	500 mesh quartz powder (g)	Curing temp. (°C)
1	100	110	6	5	30	30	3	10	50
2	100	110	6	5	30	30	3	10	70
3	100	110	6	5	30	30	3	10	85

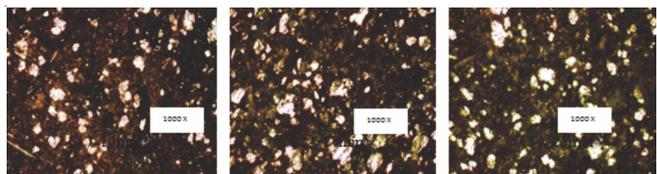


Fig. 2. Metallographic figure of material

Curing temperature

Experimental design: The bar was cured for 24 h under constant temperature of 30 °C. It was then cured for 1 h at 70 °C. An excessive temperature was shown to result in a faster reaction, sticky materials and problematic pouring in addition to sinking and stratification in the filler. Insufficient temperature was seen to lead to a slow and inadequate reaction. The optimum curing temperature for the first 24 h was thus determined at 30 °C. The optimum temperature for the final hour of high-temperature curing was supposed as 70 °C. Three group tests were performed based on the experimental formula in Table-1: 1 was cured for 1 h at 50 °C, 2 was cured for one hour at 70 °C and 3 was cured for 1 h at 85 °C.

Test data: The mechanical properties determined in the materials are shown in Table-2.

Number	Compressive strength (Mpa)	Bending strength (Mpa)	Brinell hardness
1	133.48	72.23	27.50
2	135.56	80.32	28.10
3	123.42	84.34	25.40

Analysis of test results: As shown in Table-2, an increase in material bending strength corresponded with increases in temperature. A temperature of 70 °C provided optimum compressive strength and brinell hardness as compared with 50 °C and 85 °C. In accordance, 70 °C was selected as the optimum curing temperature for the last 1 h of high-temperature curing.

Pouring process

Premixing: Due to high viscosity, thorough mixing was required of the epoxy resin, curing agent and diluent liquid to ensure consistency. Filler and mix could then be added to the mixture. The filler was mixed into the resin matrix manual using mixing tools to ensure that the powder filler was fully engaged in the vacuum injection machine [Fig. 3(1)].

Vacuum mixing and pouring: To avoid the accumulation of bubbles, an agitation speed of 90 rpm was selected for the vacuum injection machine and the materials were subsequently stirred for 25 min at 30 °C. A 30 vacuum type injection machine was used to mix the materials for 25 min with an agitation

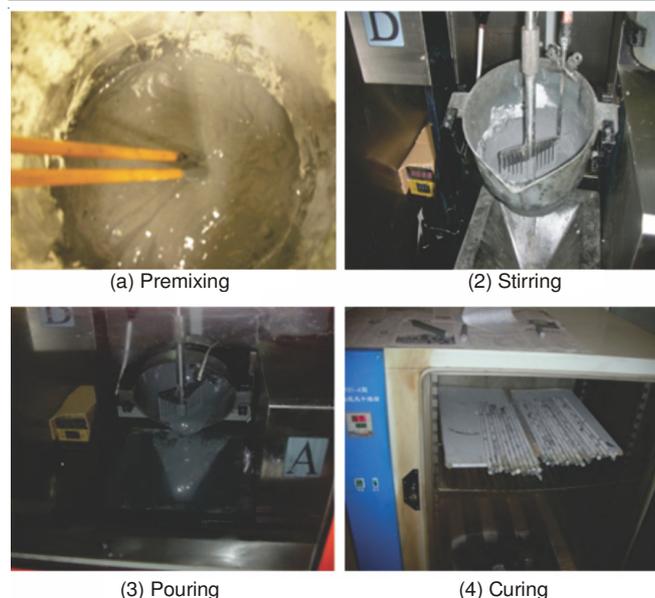


Fig. 3. Figure of premixing, stirring, pouring and curing

speed of 90 rpm to quickly discharge bubble formation. It was known that insufficient mixing time would lead to an uneven mixture while excessive mixing time would lead to extreme viscosity and uncontrollable pouring. Thus, 25 min was selected as the mixing and pouring time [Fig. 3(2)(3)].

Curing: The bar was placed in a constant temperature box and cured first for 24 h at 30 °C and then for 1 h at 70 °C. In order to achieve a smooth and full reaction, 30 °C, was chosen as the closed room temperature and a curing temperature of 70 °C for 1 h was used to complete the reaction, as shown in Fig. 3(4).

Experiment of material formula

Experimental plan: A multiple factors and levels experiment, which collocated all factors and levels, was regarded as the most comprehensive test. Although a wide-ranging test may clearly reveal internal processes, it often lacks practical application value and requires numerous tests. However, the use of an orthogonal experimental design provided an opportunity to choose a few typical combinations of factors and analyze those combinations for later implementation. The method utilized mathematical statistics for the analysis using a set of normalized orthogonal tables. Orthogonal experimental design and analysis is generally used for process optimization as part of a factorial design method⁹. An orthogonal design and a single factor experiment using a single test method were chosen to optimize material formulation¹⁰. An experimental flow chart is illustrated in Fig. 4. A strict curing process was followed. Samples that showed obvious differentiations in the mechanical properties from other samples were replaced and measured again. The mechanical properties of each sample at each level were measured at least five times and then averaged.

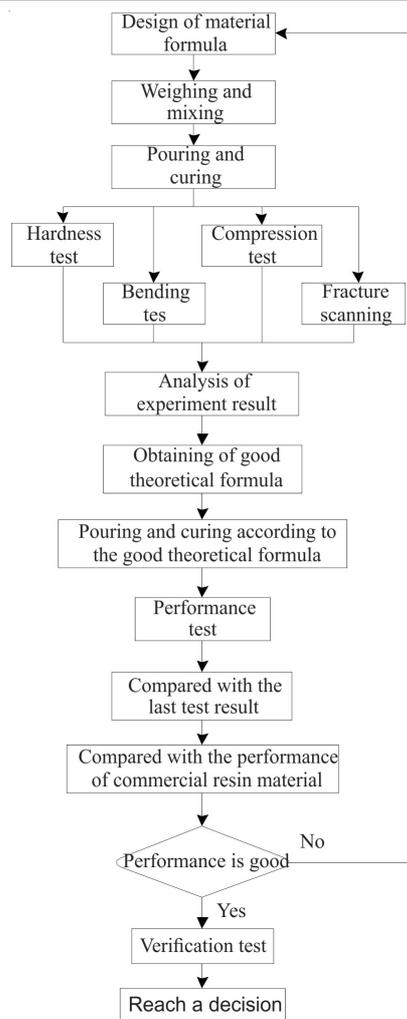


Fig. 4. Experimental flow chart

Single Factor Experiment

Epoxy resin choice

Experimental design: The effects of E44 and E51 on mechanical properties were examined using the following materials were used as a benchmark: antifoaming agent dimethyl silicone, curing agent benzene dimethylamine, reactive diluent butyl glycidyl and fillers. The fillers consisted of 600 mesh aluminum powder, 500 mesh quartz powder, 500 mesh graphite powder and short glass fiber (SGF). E44 and E51 were used with samples 1 and 2 in turn, as shown in Table-3.

Test data: The 5 test data are represented in a line chart in Fig. 5(1-3) and the material mechanics performance table is shown in Table-4 below.

Analysis of test results: The mechanical properties of 2 were far superior to those demonstrated by 1 (Table-4). Namely, E51 proved most suitable for use in composite materials and was thus selected to optimize aluminum filled epoxy resin mold material.

TABLE-3
MATERIAL FORMULA

Number	EP (g)	Quartz powder (g)	Deformer (g)	Graphite powder (g)	Aluminum powder (g)	Curing agent (g)	Reactive diluent (mL)	SGF (g)	Species of EP
1	100	15	3	10	100	16.5	15	20	E44
2	100	15	3	10	100	16.5	15	20	E51

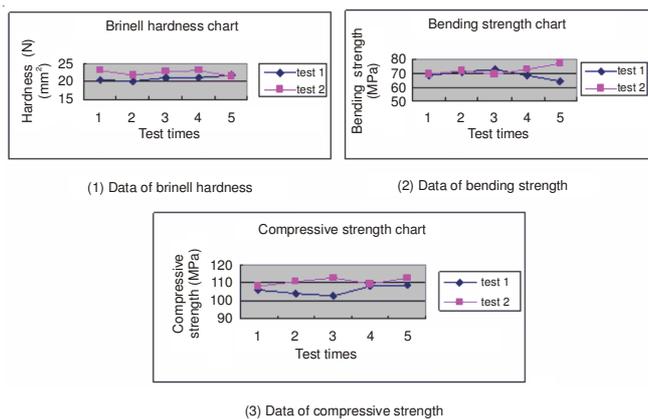


Fig. 5. Data of the mechanical performance

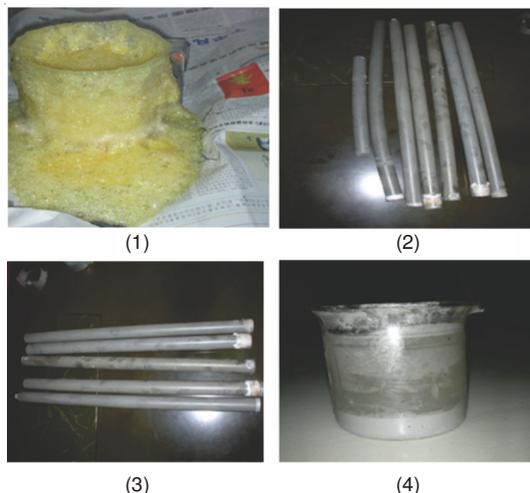


Fig. 6. Test results

Number	Brinell hardness	Bending strength (MPa)	Compressive strength (MPa)
1	20.74	69.07	105.87
2	22.40	72.38	110.70

Curing agent

Experimental design: The effects of polyamide resin 650, methyl tetrahydrophthalic anhydride and benzene dimethyl amine were compared on mechanical properties using the following materials as a benchmark: epoxy resin, defomer dimethyl silicone, reactive diluent butyl glycidyl and fillers. The fillers included 600 mesh aluminum powder, 500 mesh quartz powder, 500 mesh graphite powder and 1.5 mm SGF. In addition, polyamide resin 650, benzene dimethyl amine and methyl tetrahydrophthalic anhydride were classified as 1, 2 and 3, respectively, as shown in Table-5.

Test results: Fig. 6(1) and (2) shows the material vacuum deaeration and pouring bar of 1, respectively. Fig. 4(3) illustrates the material bar of 2, while Fig. 6(4) illustrates the mixing material 3.

Test results analysis: A vast amount of air bubbles was generated during the material vacuum defoaming of 1, as shown in Fig. 6(1). Great deformation was also seen in material 1, as shown in Fig. 5(2), due to the large shrinkage rate after curing. However, neither deformation nor bubbles appeared during the defoaming of 2 curing bar, as shown in Fig. (3). Sedimentation was seen in material 3 after mixing with fillers, as shown

in Fig. (4). The test results from 2 were seen as the most favorable and benzene dimethylamine was thus selected as the curing agent.

Diluent

Experiment design: The effects of reactive diluent butyl glycidyl ether (BGE) and non-active diluent phthalic acid dibutyl were compared on the mechanical properties using the following materials as a benchmark: epoxy resin, antifoaming agent dimethyl silicone and fillers. The fillers were 600 mesh aluminum powder, 500 mesh quartz powder, 500 mesh graphite powder and 1.5 mm SGF. Reactive diluent butyl glycidyl ether (BGE) and non-active diluent phthalic acid dibutyl were used in 1 and 2, respectively, as shown in Table-6.

Experimental results: Performance testing was conducted for each material group and repeated five times and the data was shown in Fig. 7(1-3).

Test results analysis: As shown in Table-7, the mechanical properties of 2 were significantly higher than those of 1. Moreover, instances of uneven mixing and stratification did not occur in 2. Reactive diluent butyl glycidyl ether was thus selected as diluent.

Filler

Type of filler

Experimental design: A variety of fillers, such as metal oxide, oxide and metal powder, are often used as pouring

Number	E44 (g)	Aluminum powder (g)	Curing agent (g)	Butyl glycidyl (mL)	Quartz powder (g)	Deformer (g)	Graphite powder (g)	SGF (g)	Species of curing agent
1	100	100	16.5	30	15	3	10	20	Polyamide resin 650
2	100	100	16.5	30	15	3	10	20	Benzene dimethyl amine
3	100	100	16.5	30	15	3	10	20	Methyl tetrahydrophthalic anhydride

Number	E44 (g)	Aluminum powder (g)	Curing agent (g)	Diluent (mL)	Quartz powder (g)	Deformer (g)	Graphite powder (g)	SGF (g)	Species of diluent
1	100	100	16.50	30	15	3	10	20	Non-active
2	100	100	16.50	30	15	3	10	20	Reactive

TABLE-8
TABLE OF MATERIAL FORMULA

Number	E44 (g)	Quartz powder (g)	Deformer (g)	Graphite powder (g)	Metallic filler (g)	Curing agent (g)	BGE (g)	SGF (g)
1	100	15	3	10	100 g Al	16.5	15	20
2	100	15	3	10	100 g Fe	16.5	15	20
3	100	15	3	10	100 g $Al(OH)_3$	16.5	15	20

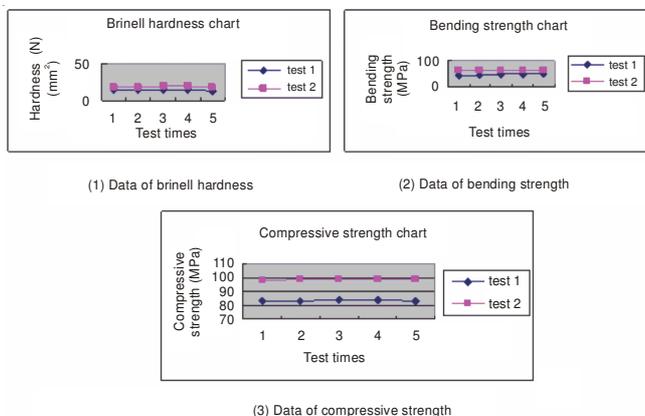


Fig. 7. Data of the mechanical performance

TABLE-7
TABLE OF MATERIAL MECHANICS PERFORMANCE

Number	Hardness (HB) (N/mm ²)	Bending strength (MPa)	Compressive strength (MPa)
1	14.07	47.69	83.32
2	18.42	65.25	98.49

materials. In this experiment, 600 mesh aluminum powder (Al), 600 mesh iron powder (Fe) and 600 mesh aluminum hydroxide powder ($Al(OH)_3$) were chosen as samples and classified as 1, 2 and 3, respectively. The material formulation is shown in Table-8.

Experimental data: The 5 test data are represented in a line chart in Fig. 8(1-3) and the average mechanical performance of the materials is shown in Table-9.

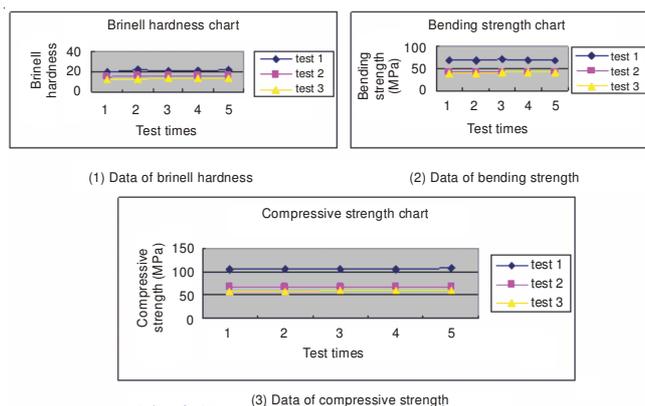


Fig. 8. Data of the mechanical performance

Experiment results analysis: As shown in Table-9, hardness, bending strength and compressive strength in material 1

TABLE-10
TABLE OF MATERIAL FORMULA

Number	E51 (g)	Al (g)	Curing agent (g)	BGE (g)	Quartz powder (g)	SGF (g)	Deformer (g)	Graphite powder (g)	Mesh of Al
1	100	110	6	5	30	30	3	10	200
2	100	110	6	5	30	30	3	10	600
3	100	110	6	5	30	30	3	10	800

was superior to that in materials 2 and 3. Moreover, stratification did not occur in 1. Aluminum powder was thus chosen as filler for rapid tooling metal.

TABLE-9
TABLE OF MECHANICAL PERFORMANCE

Number	Hardness (HB) (N/mm ²)	Bending strength (MPa)	Compressive strength (MPa)
1	20.74	69.07	105.87
2	15.92	42.37	67.34
3	13.5	40.78	59.09

Aluminum mesh

Experimental design: Metal filler aluminum powder is an essential part of epoxy resin mold material. The mesh also profoundly affects the mechanical properties of materials. A single factor experiment was thus employed for the selection of the amount of aluminum mesh. 200 mesh, 600 mesh and 800 mesh aluminum powder were used as 1, 2 and 3, respectively, as shown in Table-10.

Experimental data: The mechanical performance data of the materials is shown in Fig. 9(1-3).

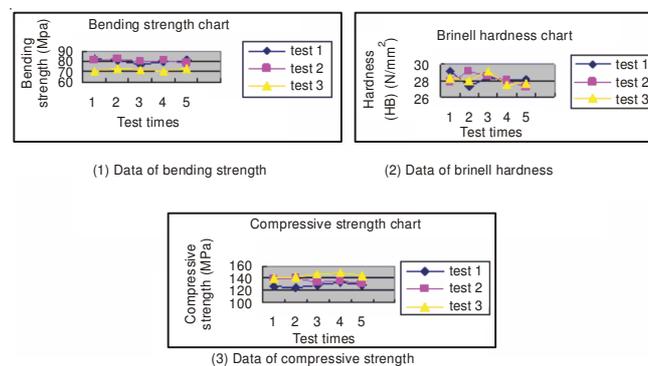


Fig. 9. Data of the mechanical performance

Analysis of experimental results: Increases in the quantity of aluminum mesh correlated with increases in compressive strength and decreases in bending strength, as shown in Table-11. However, increases in aluminum mesh showed no effect on hardness.

RESULTS AND DISCUSSION

Four factor three level orthogonal experiment

Experimental design: The orthogonal experiment was designed in accordance with a results of the single factor

Number	Compressive strength (MPa)	Bending strength (MPa)	Hardness (HB) (N/mm ²)
1	128.53	80.48	28.2
2	135.56	80.32	28.10
3	144.63	71.45	28.10

experiment. The optimum epoxy resin matrix composite material formula was defined as 600 mesh aluminum powder, benzene dimethylamine, diluent butyl glycidyl ether and 500 mesh quartz powder with 100 g E51, 30 g 1.5 mm SGF, 3 g defoaming agent dimethyl silicone, 10 g graphite powder. The four factor three level orthogonal experiment proceeded with these materials, as illustrated in Table-12. The orthogonal experimental material formula of E51 is illustrated in Table-13, which was based on the foundations of Table-12 and the base materials.

Level	Factor			
	A Al (g)	B Curing agent (g)	C BGE (g)	D Quartz powder (g)
1	100	6	5	10
2	110	9	10	20
3	120	12	15	30

Experimental results: A mechanical performance test and electron microscope scanning test were conducted for the test piece in each of the nine groups (Table-13). The material mechanical properties of each group are shown in Table-14. Relevant electron microscope scanning images are shown in Fig. 10.

Number	Hardness (HB) (N/mm ²)	Bending strength (MPa)	Compressive strength (MPa)	Density (g/cm ³)
1	26.5	72.84	120.38	1.18
2	23.4	66.43	117.18	1.21
3	23.0	65.69	107.14	1.18
4	25.8	71.87	125.25	1.24
5	23.6	63.18	110.92	1.17
6	24.2	68.65	104.52	1.25
7	24.5	67.23	105.94	1.32
8	26.1	71.03	115.44	1.29
9	22.6	68.42	104.48	1.25

Number	E51 (g)	Al (g)	Curing agent (g)	BGE (g)	Quartz powder (g)	Deformer (g)	Graphite powder (g)	SGF (g)
1	100	1(100)	1(6)	1(5)	1(10)	3	10	30
2	100	1	2(9)	2(10)	2(20)	3	10	30
3	100	1	3(12)	3(15)	3(30)	3	10	30
4	100	2(110)	1	2	3	3	10	30
5	100	2	2	3	1	3	10	30
6	100	2	3	1	2	3	10	30
7	100	3(120)	1	3	2	3	10	30
8	100	3	2	1	3	3	10	30
9	100	3	3	2	1	3	10	30

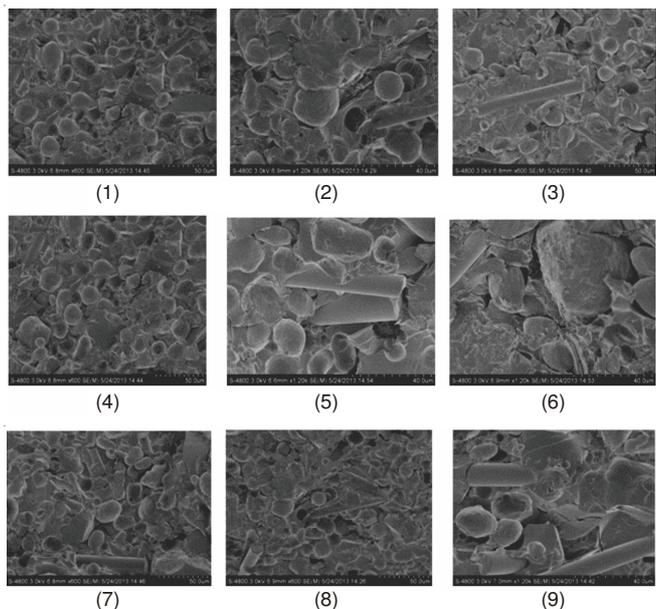


Fig. 10. Figure of 9 groups test piece of fracture morphology SEM

Analysis and discussion of experimental results: Scanning electron microscope (SEM) can be used to get various physical and chemical properties of samples which are tested without damage¹¹. We used the SEM to get the SEM graph of aluminum filled epoxy resin material and make a contrastive comparison with the results of range analysis so as to prove the validity of the range analysis.

Range analysis: The mechanical performance of each material was examined through range analysis. The optimal combination of factors was determined as shown in Table-15. R represented range analysis, which reflected the change of amplitude in test indicators. It was determined that the primary and secondary influence factors of a test index could be judged according to the size of the R¹².

Table-15 illustrates the significant effects of factor B on compressive strength and hardness. Substantial bending occurred with the application of factor C. Factor B was thus selected as the optimum factor. Factor C was selected as second choice due to its effects on the index of compressive strength, bending strength and hardness. Factors D and A were selected as third and fourth choice, respectively in accordance with their mechanical properties rankings. Finally, the primary and secondary order of factors was determined as BCDA¹³.

For factor B, performance in the later three indexes was good in level one. Level one was thus chosen. In factor C, bending strength and hardness performances were effective

TABLE-15
TABLE OF RANGE ANALYSIS

Factor	Density (g/cm)				Bending strength (MPa)			
	A	B	C	D	A	B	C	D
k ₁	1.19	1.25	1.24	1.20	68.32	70.65	70.84	68.15
k ₂	1.22	1.22	1.23	1.26	67.90	66.88	68.91	67.44
k ₃	1.29	1.23	1.22	1.24	68.89	67.59	65.37	69.53
R	0.10	0.03	0.02	0.02	0.99	3.77	5.47	2.09
Primary and secondary factors	ABCD				CBDA			
Optimal solution	A ₃ B ₁ C ₁ D ₂				C ₁ B ₁ D ₃ A ₃			
Factors	Compressive strength (MPa)				Hardness (HB) (N/mm ²)			
	A	B	C	D	A	B	C	D
k ₁	114.90	117.19	113.45	111.98	23.3	25.6	25.6	24.23
k ₂	113.56	114.51	115.64	109.21	24.53	24.37	23.93	24.03
k ₃	108.62	105.38	108.00	115.54	24.4	23.67	23.7	24.97
R	6.28	11.81	7.64	6.33	1.23	1.93	1.9	0.94
Primary and secondary factors	BCDA				BCDA			
Optimal solution	B ₁ C ₂ D ₃ A ₁				B ₁ C ₁ A ₂ D ₃			

in level one, while compressive strength performance in levels one and two was similar. Level one was thus chosen. In factor D, bending strength and compressive strength performances were good in level three, with similar hardness performances in levels two and three. Level three was thus chosen. For factor A, bending strength and compressive strength performances were similar in levels one and two. With consideration given to the main index of hardness, level two was thus chosen. Finally, the optimal combination of conditions was determined as B₁C₁D₃A₂.

Analysis of fracture morphology: As shown in Fig. 6(1-3), 1 mixing material was more compact and uniform than 2 and 3, with more complex fracture morphology. A large amount of cross-section materials and dimples were formed, which could absorb more energy during the fracture process. This was expressed through the rise in fracture energy and subsequent enhancement in toughness¹⁴. The macro performance demonstrated the improvement in mechanical properties. As seen in Fig. (2) and (3), uneven mixing and rupturing occurred in the aluminum and quartz powders due to excessive curing agent content. This could be attributed to bubble formation that prevented the aluminum powder from being completely embedded in resin or excessive filler content that led to the appearance of filler reunion. Overall, enhanced compressive strength, bending strength and hardness were seen in 1 as compared to 2 and 3. This was consistent with the material mechanical performances illustrated in Table-14.

Similar complex morphology and scattered directions in fracture morphology were displayed in Fig. 10(4) as shown in Fig. 10(1). Stress was relatively fragmented, which improved the mechanical performance of the materials. Uniform distribution was also seen in each of the samples, which suggested that the filler content was not excessive. As shown in Table-13, filler content was greater in 4 than in 2 and 3. As filler content was not excessive in 2 or 3, it was deduced that the inferior material mixing seen in 2 and 3, as compared to 1, was due to an excess of curing agent content. Level 1 was thus chosen for the curing agent content. It was originally supposed that an increase of aluminum powder and quartz powder in 4 would lead to enhanced mechanical performance. However, as shown in Table-14, 1 was found superior to 4 in material

hardness and bending strength. As both 1 and 4 utilized the same pouring process, the differences in mechanical performance between 4 and 1 were evidently due to one or more content factors. As shown in Table-13, diluent content was higher in 4 than in 1. The inferior hardness and bending strength of 4 was therefore attributed to diluent content. The optimum diluent content was thus determined as level 1. The functionality of reactive diluent in epoxy resin has a tendency to inhibit chain generation, thus hindering improvements in the performance of mechanical properties. As a result, material brittleness increases which subsequently reduces material hardness and bending strength in the curing system of the macro trend¹⁵. This rationale corresponds with the fracture morphology analysis outlined above. Fig. (5) and (6) display lower density as compared with Fig. (4). This would suggest that excessive curing agent content in 5 and 6 led to accumulation of bubbles, while high filler content also led to gathering. As illustrated in Table-13, lower filler content was seen in 5 and 6 as compared with 4. As the filler content was low, the accumulation of bubbles in 5 and 6 was attributed to excessive curing agent. Mechanical performance was thus most favorable in 4 and the optimum curing agent level was determined as level 1. As shown in Fig. (7), the uniform material mixing and compactness of 7 indicated that curing agent and filler levels were not excessive. However, gathering was shown to lead to material cracking, which had serious implications for connections between resin and filler, as shown in Fig. (8) and (9). These weak material links deteriorated under stress, indicating an excess of curing agent and filler in 8 and 9. However, as shown in Table-14, 8 demonstrated superior mechanical performance when compared to 7 which was attributed to higher diluent content in 7.

In conclusion, equivalent results were seen in fracture morphology and range analysis.

Conclusion

Following comprehensive analysis, the optimal combination was determined as B₁C₁D₃A₂. More specifically, this comprised of a mass fraction including 37.41 % of E51 resin, 37.41 % of 600 mesh aluminum powder, 2.04 % of curing agent benzene dimethylamine, 2.04 % of reactive diluent butyl

glycidyl ether, 10.2 % of 500 mesh quartz powder, 10.2 % of 1.5 mm short glass fiber, 1.02 % of antifoaming agent dimethyl silicone oil and 3.4 % of 500 mesh graphite powder. However, the optimal combination formula was not among the nine tests. The material mechanical performance of the optimal combination formula was verified, with the test repeated five times, as shown in Table-16.

TABLE-16
TABLE OF MATERIAL MECHANICAL PERFORMANCE

Compressive strength (MPa)	Brinell hardness (HB) (N/mm ²)	Bending strength (MPa)
135.56	28.1	80.32

The performance of the international commercial CW series tooling resin, Araldite brand, from the Switzerland Ciba Specialty Chemicals company is illustrated in Table-17¹⁶.

TABLE-17
TABLE OF MATERIAL MECHANICAL PERFORMANCE OF CIBA

Compressive strength (MPa)	Shore hardness (HS)	Bending strength (MPa)
142	88	75

The test material showed similar compressive strength and practical application as Ciba, as shown in Tables 16 and 17. The test material demonstrated slightly lower compressive strength but higher bending strength, as compared to Ciba.

ACKNOWLEDGEMENTS

The authors gratefully acknowledged the fund support rendered by Xi'an Jiaotong University for the topic which is the open subject of Mechanical Manufacturing Systems Engineering State Key Laboratory (Grant Number: sklms2011002).

REFERENCES

1. Y.Y. Quan and L.Z. Zhang, *Appl. Surf. Sci.*, **292**, 44 (2014).
2. J.L. Song, Y.C. Jin, G.S. Fu, A.J. He and W.J. Fu, *J. Appl. Polym. Sci.*, **124**, 1744 (2012).
3. W. Zhang, L.T. Kuhn, P.S. Jørgensen, K. Thydén, J.J. Bentzen, E. Abdellahi, B.R. Sudireddy, M. Chen and J.R. Bowen, *Microsc. Microanal.*, **19**, 501 (2013).
4. D. King and T. Tansey, *J. Mater. Process. Technol.*, **121**, 313 (2002).
5. J.H. Zhao and J. Zhang, *Eng. Plastics Appl.*, **35**, 47 (2007).
6. K. Nagahanumaiah, K. Subburaj and B. Ravi, *Comput. Ind.*, **59**, 262 (2008).
7. Y.G. Wang, *Rapid Tooling and Its Applications*, Huazhong University of Science and Technology Press, Wuhan (2003).
8. J.P. Sun. *Experimental Research on Materials Components and Process of Metal Resin Mold*, Shandong University Press, Shandong (2005).
9. R.J. Liu, Y.W. Zhang, C.H. Wen and J. Tang, *Test Technol. Manag.*, **27**, 52 (2010).
10. L. Liu, Z.X. Xin, *Rubber/Plastic Technol. Equipment*, **30**, 8 (2004).
11. V.G.M. Sivel, J. Van Den Brand, W.R. Wang, H. Mohdadi, F.D. Tichelaar, P.F.A. Alkemade and H.W. Zandbergen, *J. Master Process Technol.*, **214**, 237 (2004).
12. G. Yang, Y.G. Zhou, Y.D. Zhang and Y.F. Yue, *Mater. Sci. Eng.*, **35**, 60 (2012).
13. L.J. Pan and J.Q. Chen, *Experiment Design and Data Processing*, Southeast university press, Nanjing (2008).
14. R.L. Zhang, Y.S. Liu and Y.X. Jin, *Mater. Des.*, **12**, 71 (2011).
15. J. Zhang, *Urban Construction and Commercial Outlets*, **42**, 287 (2009).
16. J. Zhang, *Study on Rapid Prototyping Material Based on Epoxy Resin*, Chongqing University, Chongqing, China, p. 19 (2007).