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Microstructure, Mechanical Properties, and Age-Hardening Behavior of an Al-Si-Fe-Mn-Cu-Mg Alloy Produced by Spray Deposition

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It has been recognized generally that the spray-deposited process is an innovative technique of rapid solidification. In this paper, Al-20Si-5Fe-3Mn-3Cu-1Mg alloy was synthesized by the spray atomization and deposition technique. The microstructure and mechanical properties of the spray-deposited alloy were studied using x-ray diffraction, scanning electron microscopy, transmission electron microscopy (TEM), and tensile tests. It is observed that the microstructure of spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg alloy is composed of the α-Al,Si and the particle-like $Al_{15}(FeMn)_3Si_2$ compounds. The aging process of the alloy was investigated by microhardness measurement, differential scanning calorimetry analysis, and TEM observations. The results indicate that the two types of precipitates, S-Al_2CuMg and σ-$Al_5Cu_6Mg_2$ precipitate from matrix and improve the tensile strength of the alloy efficiently at both the ambient and elevated temperatures (300 °C).

Keywords age-hardening behavior, mechanical properties, microstructure, spray deposition

1. Introduction

The stringent requirements of the automotive and aerospace industries have prompted the design of advanced materials with high-strength-to-weight ratio, low coefficient of thermal expansion, and high-wear resistance (Ref 1, 2). The low coefficient of thermal expansion and good wear resistance characteristic of hypereutectic Al-Si alloy have engendered considerable interest in this alloy family as candidate materials for automotive, electrical, and aerospace applications (Ref 3, 4). Unfortunately, ingot metallurgy aluminum alloys in the hypereutectic Si regime exhibit poor mechanical properties. The slow cooling rate conditions typical of the ingot metallurgy process give rise to coarse primary Si crystals in combination with a high volume fraction of interdendritic eutectic phase rendering inherent brittleness (Ref 5). Rapid solidification processing has been used to improve the strength level of hypereutectic Al-Si alloys. Cylindrical-shaped free-standing bulk component of hypereutectic Al alloy with nanostructured grains was fabricated using vacuum plasma spray forming technique by Laha et al. (Ref 6). Powder metallurgy techniques provide a means of fabricating hypereutectic Al-Si alloys (Ref 7). However, limitations of the technique include relatively higher cost, complicated processing steps, longer production cycles, or specific protecting atmosphere. In contrast to the powder metallurgy and vacuum plasma spray technique, spray deposition process is believed to be an effective technique because of the advantages of higher production rate, low inherent oxide contents, and near-net products. Spray deposition is a novel process which is used to manufacture rapidly solidified bulk and near-net-shape preforms. The process consists of two consecutive steps, namely atomization of a liquid metal into droplets and deposition of the droplets into a three-dimensional preform (Ref 8, 9). Spray deposition process has an obvious modification in size, morphology, and distribution of the primary Si phase in the hypereutectic Al-Si alloy.

In order to improve the mechanical properties of hypereutectic Al-Si alloys, a modification in composition is necessarily made by adding alloying elements such as Cu, Mg, Fe, Mn to this alloys (Ref 10, 11). Cu and Mg promote strengthening through solution and precipitation hardening. Fe and Mn improve elevated mechanical properties through formation of intermetallic phases that are relatively stable at temperature of up to 300 °C.

Precipitation strengthening in age-hardenable aluminum alloy is generally due to the formation of precipitates during aging treatment. In this study, the age-hardening behavior of spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg alloy was investigated, and the effect of the age-hardening phases on the tensile strength of the alloy was analyzed.

2. Experimental Procedures

The nominal composition of the alloy was: 20% Si, 5% Fe, 3% Mn, 3% Cu, 1% Mg (wt.%), and balance aluminum. The spray deposition experiments were conducted in an environmental chamber. During spray-deposition process, the molten

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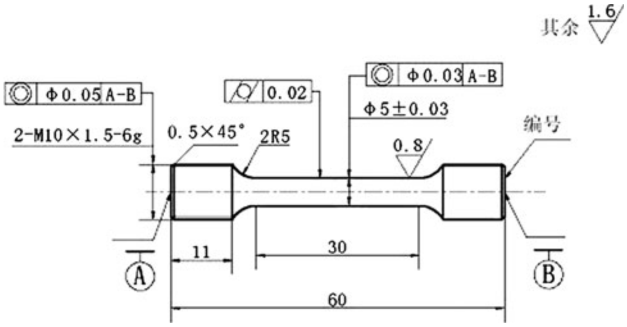

Fig. 1 Diagram of the samples for tensile test

metal was atomized by N_2 at 800 °C, the distance of atomizing deposition was kept constant at 400 mm. The preform, after being cut into cylindrical billets was extruded at a temperature of 400 °C with a reduction ratio of 10:1 and a ram speed of 5 mm/s. After extrusion, the materials were cooled in air. In this study, a commercially used scheme of heat treatment for the alloy was applied, which involved solution treating at 470 °C for 1.5 h followed by water quenching, and artificial aging at 135 °C for 4, 8, 12, 16, 20, 24, 28, 32, and 48 h.

The rectangular pieces were cut from the water quenched sample and investigated by differential scanning calorimetry (DSC). A Dupont 2100 instrument was used in a nitrogen atmosphere with a heating and cooling rate of 10 K/min. A LECO M-400 microhardness machine was used for hardness measurement with a load 50 g applied for 10 s. The microstructures of spray-deposited materials were characterized using scanning electron microscopy (SEM), transmission electron microscopy (TEM), and x-ray diffraction (XRD). A S360-type SEM working at 15 kV was used to observe the microstructures. The SEM samples were prepared using standard metallographic techniques and were etched using Keller's reagent. The TEM studies were conducted on a H-800-type transmission electron microscope at an acceleration voltage of 200 kV. The XRD experiments were performed on a Japan Rigaku diffractormeter using Cu K_α radiation. The room and elevated temperature tensile tests were conducted with an Instron tensile testing machine at an initial strain rate of 0.5 s^{-1}. The elevated temperature tensile tests were conducted at 573 K at the same initial strain rate. Three samples were used during each test. Figure 1 shows the diagram of the samples for tensile test.

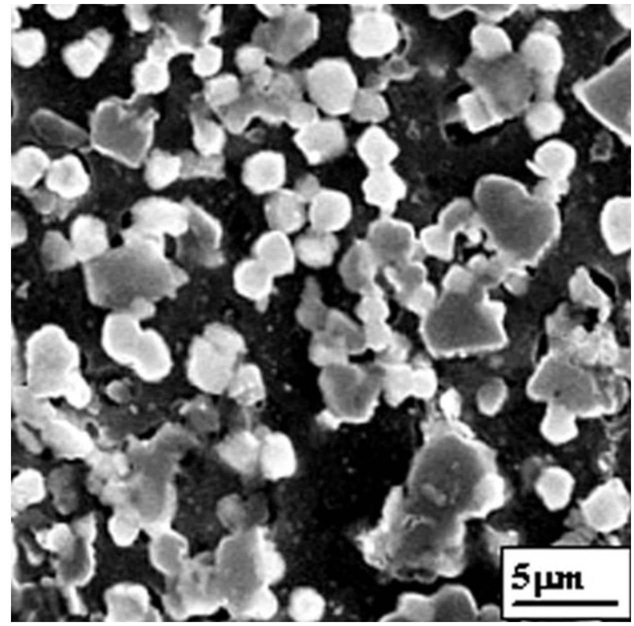

Fig. 2 SEM micrograph of the spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg alloy

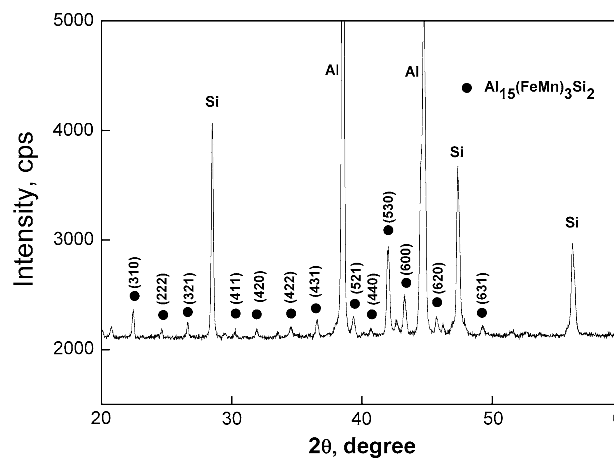

Fig. 3 X-ray diffraction pattern of the spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg alloy

3. Results and Discussion

3.1 Microstructure

Figure 2 shows the typical SEM microstructure of the spray-deposited alloy, which is composed of the aluminum matrix, the silicon phase mostly with a spherical particle shape, and some fine intermetallic phases. The intermetallic phases are about 1-3 µm in size. The particle-like intermetallic phase is distributed randomly in the microstructure. Energy dispersive x-ray (EDX) analysis was used to check the composition of the intermetallic phases with a spherical particle shape, it showed that the particle-like compound contains Al, Si, Fe, and Mn.

Figure 3 shows the XRD pattern of the spray-deposited alloys. XRD was performed to identify second phases, and analysis of the diffraction patterns shows that the particle-like compounds are $Al_{15}(FeMn)_3Si_2$ phases. Figure 4(a) shows the TEM photograph of the spray-deposited alloy. EDX analysis showed that intermetallic phases with a particle shape contain elements of Al, Si, Fe, and Mn, and this phase being identified as $Al_{15}(FeMn)_3Si_2$ compound using electron diffraction, as can be seen in Fig. 4(b). The TEM results are in good agreement with the XRD ones, as shown in Fig. 3.

3.2 Aging Process

3.2.1 Precipitation Hardening Response. Samples of the spray-deposited alloy were solution heat treated at 470 °C for 1.5 h followed by water quenching. These samples were then aged for various times at 135 °C. The resulting aging curve is shown in Fig. 5. Data points in Fig. 5 show that two

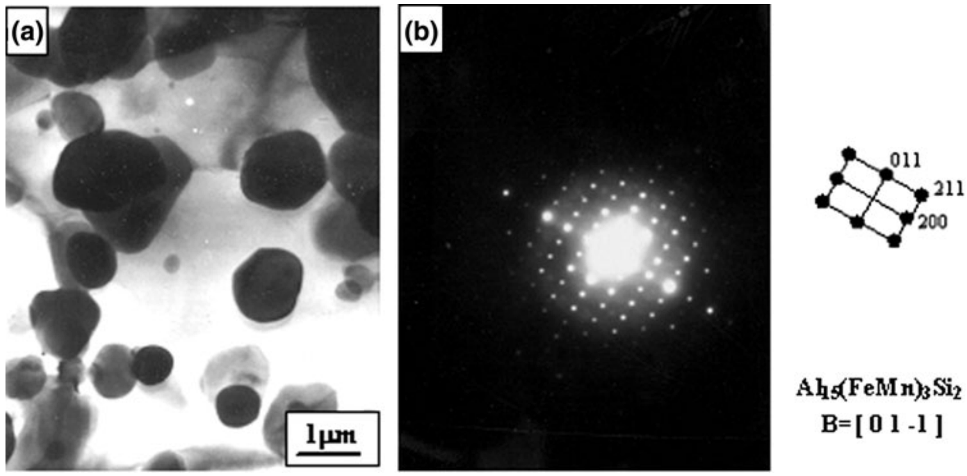

Fig. 4 TEM micrograph of spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg alloy showing the presence of the $Al_{15}(FeMn)_3Si_2$ phase: (a) bright-field image and (b) selected area diffraction pattern

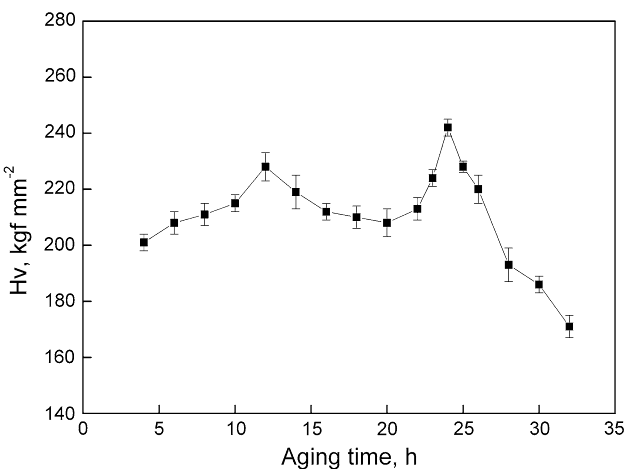

Fig. 5 Microhardness of spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg alloy at 135 °C for various aging time

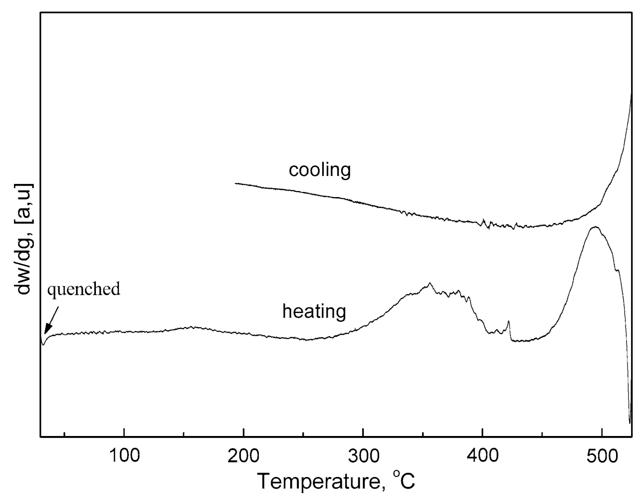

Fig. 6 DSC thermogram for the specimen of spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg alloy

hardness peaks appeared, which indicate that two precipitating phases may precipitate from matrix. The peak hardness values achieved at an aging temperature of 135 °C were 232 and 252, and corresponding times to reach these peak hardness values were 12 and 24 h, respectively.

3.2.2 Differential Scanning Calorimetry. To realize the aging behavior of the alloy during an aging treatment, the solution-treated specimen was made into DSC sample. Figure 6 shows a typical DSC thermogram for the specimen of the alloy. It can be seen that two exothermic peaks appear during heating, but no endothermic reaction occurs during cooling process. The absence of an endotherm following the GP zone formation peak at low temperature suggests that the strengthening phases are directly precipitate from the matrix. The reasons were as follows: the cooling rate was very high during spray deposition, and the solid solubility of solute atoms was increased; therefore, the precipitation rate of the alloy element became faster. Furthermore, the amount of Si, Fe, and Mn in the alloy also changes the aging characteristic of Al-Cu-Mg alloy, which may prevent the precipitation of GP zone also.

3.2.3 TEM Observation. Figure 7(a) shows the TEM micrograph of the alloy aged at 135 °C for 12 h, it can be seen that the some small precipitates were presented in the microstructure. Higher magnification by TEM of the spray-deposited alloy aged at 135 °C for 12 h (Fig. 8a), revealed the precipitates have a cubic morphology. Indexing of the selected area diffraction pattern indicates that the cubic-like phase is σ-$Al_5Cu_6Mg_2$ compound, as can be seen in Fig. 8(b). Figures 9 and 10 shows the microstructures of the alloy aged at 135 °C for 48 h, selected area diffraction analysis indicated that the plate precipitate was S-Al_2CuMg, and the cubic precipitate was σ-$Al_5Cu_6Mg_2$.

According to the results of microhardness measurement, DSC, TEM, and SAD, it can be seen that the two types of precipitates appeared during aging of the alloy at 135 °C. In quenched alloy, the first stage of hardening has been interpreted to be due to σ-$Al_5Cu_6Mg_2$ formation. After aging for 24 h, which is close to the second stage of hardening, DSC combined with TEM methods have shown that the second hardening peak corresponds to the completion of S-Al_2CuMg formation.

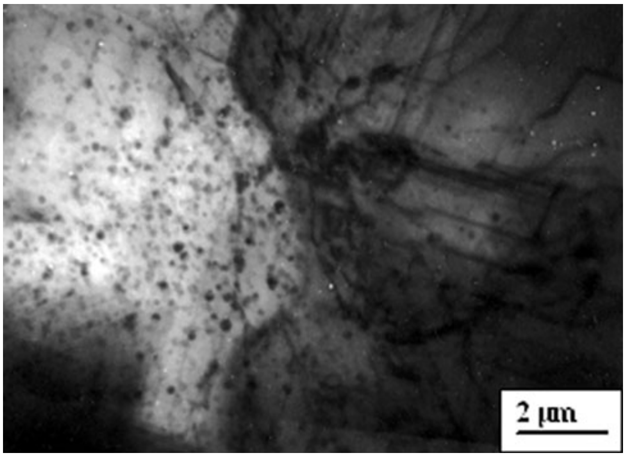

Fig. 7 TEM of spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg alloy aged at 135 °C for 12 h

The absence of an endothermic reaction following the GP zone formation peak suggests that S-Al_2CuMg and σ-$Al_5Cu_6Mg_2$ are directly precipitate from the matrix, which is associated with rapid solidification and Si, Fe, and Mn additions. Therefore, the higher hardness of the alloy after aging at 135 °C for 24 h can be attributed to the precipitation of those two precipitates.

3.3 Mechanical Properties

Spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg and Al-20Si-5Fe-3Mn alloys were subjected to tensile testing at room and elevated temperature (300 °C). Table 1 summarizes the tensile test results of spray-deposited and vacuum plasma spray formed hypereutectic Al-Si alloys. It is worth noting that the tensile strengths of Al-20Si-5Fe-3Mn-3Cu-1Mg alloy both at room and elevated temperatures displayed superior than Al-20Si-5Fe-3Mn alloy. Obviously, S-Al_2CuMg and σ-$Al_5Cu_6Mg_2$ precipitates play an important role for the

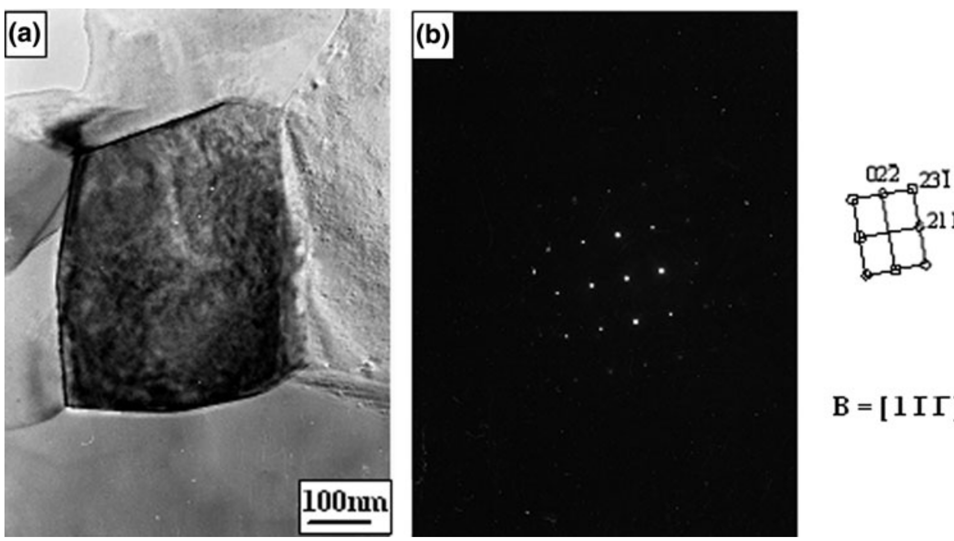

Fig. 8 TEM micrograph of spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg alloy aged at 135 °C for 12 h: (a) bright-field image and (b) SAD pattern

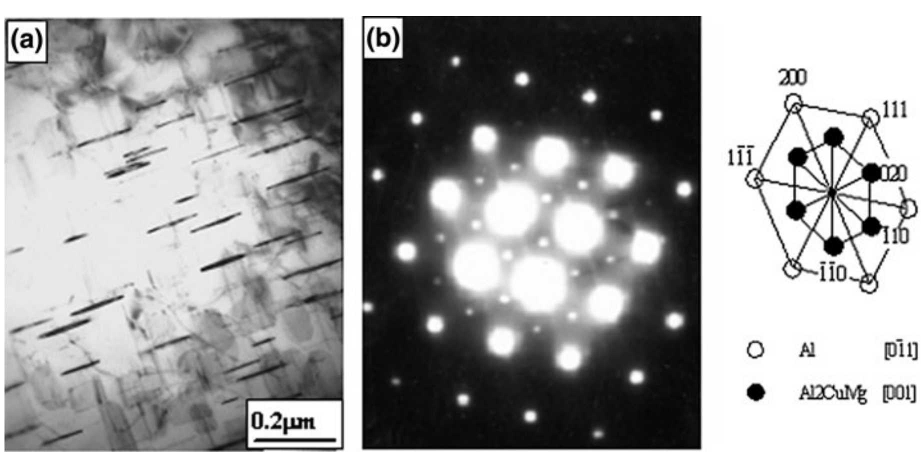

Fig. 9 TEM micrograph of spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg alloy aged at 135 °C for 48 h: (a) bright-field image of the plate precipitate and (b) SAD pattern

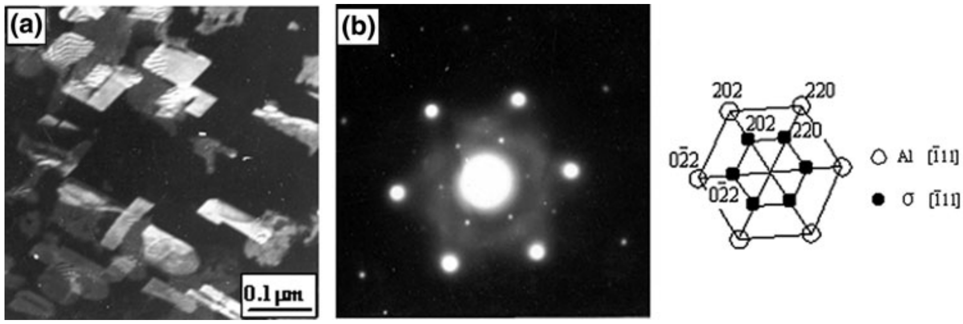

Fig. 10 TEM micrograph of spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg alloy aged at 135 °C for 48 h: (a) bright-field image of the cubic precipitate (b) SAD pattern

Table 1 Tensile properties of spray-deposited and vacuum plasma spray formed alloys

Alloy	Processing	Temperature, °C	YS, MPa	UTS, MPa
Al-20Si-5Fe-3Mn	SD + 400 °C Extr. + 470 °C temper	25	210	318
		300	148	262
Al-20Si-5Fe-3Mn-3Cu-1Mg	SD + 400 °C Extr. + T6 temper	25	328	452
		300	276	324
Al-21Si-2Ni-1Cu (Ref 6)	Vacuum plasma spray	25	···	345

SD, Spray deposition; Extr., extrusion; T6 temper, 470 °C/1.5 h + 135 °C/24 h; YS, yield strength; UTS, ultimate tensile strength

increasing of the tensile strength. A lot of dispersion-strengthened S-Al_2CuMg precipitates distributed in the matrix of Al-20Si-5Fe-3Mn-3Cu-1Mg alloy, they can prevent dislocation moving, and enhance strength of the alloy at room temperature. Moreover, the plate of S-Al_2CuMg precipitate coarsens significantly at temperature higher than 150 °C causing loss of mechanical properties, while the cubic σ-$Al_5Cu_6Mg_2$ precipitate was determined to be stable to a temperature of 400 °C (Ref 12), at which it will dissolve into matrix. The dispersion of the thermally stable cubic σ-$Al_5Cu_6Mg_2$ precipitates, in combination with their inhibition to growth of the grains occurring at elevated temperature, is considered to be the key to the enhanced hot strength of the alloy. The tensile test results indicate that the spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg alloy has better strength than the vacuum plasma spray processed Al-21Si-2Ni-1Cu alloy.

4. Conclusion

The microstructure of spray-deposited Al-20Si-5Fe-3Mn-3Cu-1Mg alloy is composed of the Al matrix, Si phase and particulate-like $Al_{15}(FeMn)_3Si_2$ compound. After aging treatment at 135 °C, the two types of precipitates, plate S-Al_2CuMg and cubic σ-$Al_5Cu_6Mg_2$ precipitates from matrix and improve the tensile strength of the alloy efficiently at both the ambient and elevated temperatures (300 °C).

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