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INFLUENCE OF IRON AND NICKEL ADDITIONS ON THE CONDUCTIVITY, MICROHARDNESS AND MICROSTRUCTURE OF PURE ALUMINUM

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The effects of Fe and Ni additions on the electrical conductivity, thermal conductivity, microhardness and microstructure of pure Al was investigated. Both Fe and Ni have low solubility in solid Al and therefore form hard intermetallic phases, which are beneficial for producing strong, conductive alloys for electrical and thermal design requirements. Pure Al was melted at 730 °C, and 0.6, 1, 1.5 and 3 wt.% Fe or Ni was added and stirred into the melt before pouring into a steel mold. With increasing Fe content, the castings showed increases in microhardness and contained Al-Fe intermetallic phases with blocky as well as needle morphologies. With the addition of Ni, the formation of Al_3Ni intermetallic phases increased alloy hardness. On a per weight percent basis, Fe additions were found to add 3.9 HV/wt.% to microhardness and reduce electrical conductivity by 3.4 % IACS/wt.%. For Ni addition, the increase in microhardness and decrease in electrical conductivity were 4.3 HV/wt.% and 1.7 % IACS/wt.%, respectively. Both Fe and Ni additions show potential for the production of cast Al alloys that are both strong and conductive.

Introduction

Lightweighting and electrification are important strategies for automotive manufacturers aiming for the reduction of carbon emissions and improvement of fuel economies. Aluminum and its alloys have a unique advantage over other lightweight materials, such as polymers, carbon fibre-polymer composites and advanced steels, in that Al alloys have high strength-to-weight ratios as well as high thermal and electrical conductivity (TEC). Pure Al has higher TEC than its alloys because of the lack of impurities in solid solution that act as scattering centers [1], but its strength is too low to be used for most practical applications. Wrought 6000 series (Al-Si-Mg) alloys typically have high TEC (~50 % IACS, ~200 W/m-K) and high yield strengths (> 150 MPa). This is due to their compositions having a low concentration of alloying elements, and the combination of Si and Mg forming hard Mg_2Si precipitates [2]. However, it is difficult to use wrought alloys to form complex geometries, which necessitates the development of enhanced cast components. Cast Al-Si alloys have reasonable strength (>150 MPa yield strength) but lower TEC (100-130 W/m-K) than the wrought alternatives. This is partially due to the presence of high concentrations of Si (>5 wt.%) [1] in most cast industrial (3xx series) Al alloys. Therefore, there is a need for castable, high TEC alloys to produce geometrically-complex components for automotive, electronics and lighting applications.

Recent research to develop castable and conductive materials has included the development of new alloys based on low contents of Si, Cu, Mg, Fe and Ni [3]. Alloys based on the Al-Fe and Al-

Ni systems show potential for producing castable, high TEC materials with moderate strength. These alloys have short freezing ranges, and a high fraction of their solid formation occurs in a eutectic reaction, similar to the solidification characteristics of Al-Si alloys. Also, the Al-Fe and Al-Ni eutectic compositions are ~1.8 wt.% Fe and ~6 wt.% Ni, respectively, which is much lower than the 12 wt.% Si eutectic composition for the Al-Si system [4]. Alloy additions at or near the eutectic composition result in alloys with short freezing ranges, a property that is advantageous for castability. Therefore, Al-Fe and Al-Ni alloys with compositions near their eutectic composition would have short freezing ranges, high castability and high TEC since only a small amount of alloy elements as compared to Al-Si alloys are required to reach their respective eutectic compositions.

The solubility of Ni and Fe in Al are each 0.05 wt.%, which enables almost all of the alloying elements to be incorporated into high strength intermetallic phases [3]. The formation of Al-Fe intermetallic phases are estimated to increase the hardness by 4.16 HV/wt.% Fe and decrease the electrical conductivity by 1.4% IACS/wt.% Fe when added up to 1.5 wt.% [5]. It has been found that Si and Fe can improve fluidity and strength [6], such that it is possible to produce Al-Fe-Mg-Si alloys showing fluidity comparable to Al ADC12 (equivalent to A383) [3]. In addition, Fe is present as an impurity in Al alloys or intentionally added to prevent mold erosion/mold sticking [7]. When used in combination with Si, adding Fe can form fine Al-Fe-Si particles if subjected to a homogenization (>570°C for multiple hours) heat treatment [8]. Additions of Ni have been shown to enhance alloy strength by the formation of thin and homogeneous Al_3Ni which prevents crack growth and increases strength [9,10]. The combination of Ni and Fe results in Al_9FeNi hard, intermetallic phases [11,12].

Present Research

The objective of this research was to examine the changes in TEC, microhardness and microstructure of pure Al with Fe and Ni addition. Both Al-Fe and Al-Ni systems show promise as being the basis for castable, high strength and high TEC alloys for emerging heat dissipation applications.

Experimental Procedure

Melting and Casting

Binary alloys of Al-0.6, 1, 1.5, 3 wt.% Fe and Al-0.6, 1, 1.5, 3 wt.% Ni were prepared in an electric resistance furnace, using pure Al (99.88 wt.%), Fe (99 wt.%) and Ni (99.6 wt.%) wires. A boron nitride coating was applied to all steel tools including a dross skimmer, stirring rod, and closed steel mold. To prepare an Al-Fe alloy, 200 g of pure Al was melted in a clay graphite crucible and heated to 730 °C. Once at temperature, the Al melt was skimmed using a steel dross skimmer. The desired amount of Fe was added to the melt and manually stirred with a steel rod for 30 seconds. The melt was then held at 730 °C for 30 minutes before stirring once more for 30 seconds. The melt was poured at 720 °C into a steel permanent closed mold, preheated to 300 °C. The permanent mold produced a cylindrical disk 12 mm thick and 65 mm in diameter. The preparation of the Al-Ni alloys was similar to the Al-Fe alloys, except the Ni was added and stirred while the melt was protected by an Ar cover gas flowing at 4.7 L/min. The Al-Ni melts were poured immediately after stirring with no holding time. A minimum of two samples were produced for each condition, and the alloy compositions were checked using the average of at least five measurements of a Oxford Instruments Foundry-Master Pro optical emission spectrometer (OES).

Scanning Electron Microscopy

The samples for scanning electron microscopy were sectioned from the internal surface along the largest diameter of the cylindrical disks. Samples for microscopy were prepared using successively finer SiC grinding cloths (120, 320, 600 and 1200 grit) and polishing steps (9, 3 and 1 μm) with a final polish of 0.05 μm colloidal silica. Scanning electron microscopy (SEM) was conducted using a JEOL JSM-6380LV SEM operating at 20 keV, with an energy-dispersive X-ray spectroscopy (EDX) attachment.

Electrical and Thermal Conductivity Measurements

Electrical conductivity measurements were conducted using an EtherNDE Sigmacheck 2 eddy current conductivity device, using a 13 mm probe operating at 60 Hz. An average of five measurements were taken at various locations per sample. Thermal conductivity was calculated by converting electrical conductivity measurements into thermal conductivity values, using the Wiedemann-Franz Law [13].

Microhardness

Microhardness measurements were conducted using a Clemex CMT 8.0 microhardness tester with Clemex CMT image analysis software. The microhardness measurements were conducted using a load of 50 g and a dwell time of 10 seconds. The average microhardness was calculated using 20 indents made in a cross pattern along the sample, with a 200 μm centre-to-centre distance between each indent.

Results and Discussion

Composition Analysis

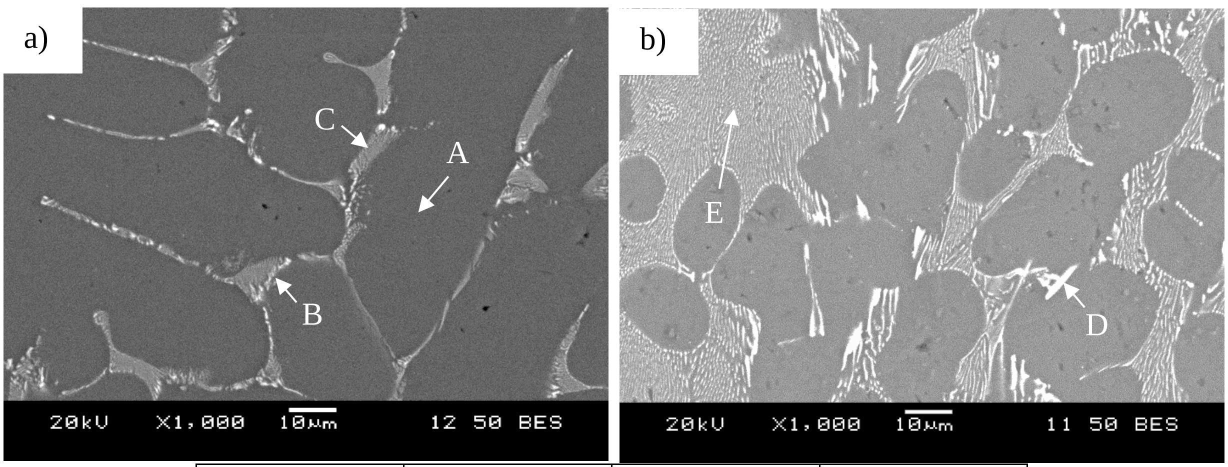
The measured compositions of the prepared Al-Fe and Al-Ni alloys collected using the OES are shown in Table 1. The target compositions were achieved while maintaining very low concentrations of Cr, Mn, Ti and V, which are highly detrimental to TEC. The electrical and thermal conductivity measurements obtained from the prepared alloys were therefore reflective of the Fe or Ni addition and likely not influenced by the presence of other impurities.

Table 1: Average Composition of Prepared Al-Fe and Al-Ni Alloys

Alloy Condition	**Al (wt.%)**	**Fe (wt.%)**	**Ni (wt.%)**	**Si (wt.%)**	**Mg (wt.%)**	**Cu (wt.%)**	**Zn (wt.%)**	**Sum of Cr, Mn, Ti and V (wt.%)**
Pure Al	Bal.	0.066	0.007	0.003	0.032	0.002	0.002	0.011
Al+0.6Fe	Bal.	0.573	0.007	0.003	0.032	0.007	0.007	0.014
Al+1.0Fe	Bal.	0.924	0.011	0.005	0.043	0.004	0.008	0.014
Al+1.5Fe	Bal.	1.480	0.007	0.003	0.085	0.007	0.012	0.015
Al+3.0Fe	Bal.	2.803	0.008	0.004	0.037	0.009	0.008	0.016
Al+0.6Ni	Bal.	0.071	0.612	0.005	0.058	0.003	0.015	0.014
Al+1.0Ni	Bal.	0.065	1.007	0.003	0.033	0.001	0.004	0.011
Al+1.5Ni	Bal.	0.064	1.404	0.005	0.041	0.003	0.005	0.013
Al+3.0Ni	Bal.	0.059	3.223	0.005	0.030	0.001	0.001	0.010

Microstructure of Al-Ni and Al-Fe Alloys

The microstructures of the Al-Ni and Al-Fe alloys were examined using SEM and are shown in Figure 1 and Figure 2 respectively. The pure Al microstructure only contained an Al matrix, with no noticeable intermetallic phases present. Figure 1a is the microstructure of the Al--1.5 wt.% Ni sample with corresponding EDX analysis for the three phases observed. Point A is the Al matrix which, was nearly pure Al. The solid solubility of Ni in Al is 0.05 wt.%, and so nearly all the added Ni formed intermetallic phases with the Al. Two types of Al-Ni intermetallic phases were observed, as seen in EDX points B and C. The presence of Fe in points B and C was likely as an impurity and not actually a significant contributor to the observed microstructure. Globular type Al-Ni particles can be observed at point B and fibrous Al-Ni particles at point C. Both show stoichiometries corresponding to Al_3Ni, similar to what was observed by previous researchers [11,12]. The Al-3 wt.% Ni alloy in Figure 1b is similar to the microstructure of the Al-1.5 wt.% Ni alloy in Figure 1a except with a larger proportion of both blocky and fibrous Al_3Ni. The thin Al_3Ni has been observed by other researchers [14] and increased Ni addition also resulted in much larger Al_3Ni blocky particles as shown in point D.

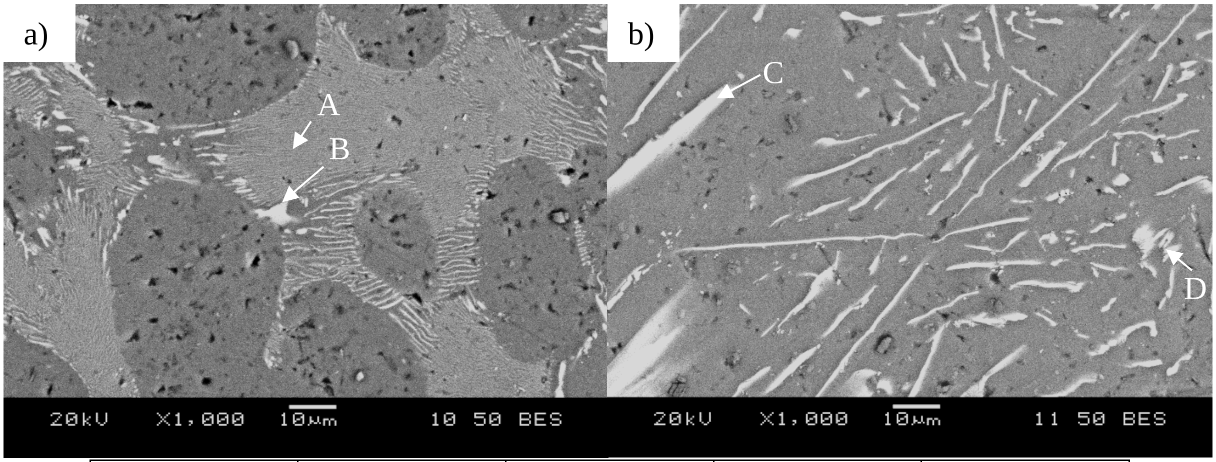


Location	Al (wt.%)	Fe (wt.%)	Ni (wt.%)
A	100.0	-	-
B	91.7	0.6	7.7
C	92.0	0.7	7.3
D	72.9	-	27.1
E	93.8	-	6.2

Figure 1: SEM Images of a) Al-1.5 wt.% Ni and b) Al-3 wt.% Ni with Corresponding EDX Spot Analysis Results of Intermetallic Phases

The microstructure of Al-1.5 wt.% Fe and Al-3 wt.% Fe are shown in Figure 2a and b, respectively. The Al-Fe alloys also showed that the Al matrix consists of nearly pure Al, given that all of the Fe present was out of solution and formed intermetallic phases. The small, black particles embedded in the soft Al matrix shown in Figure 2a are from the 0.05μm colloidal silica suspension used in the final polishing procedure. In the Al-1.5 wt.% Fe alloy (Figure 2a), fibrous Al-Fe eutectic phases were present. When the Fe concentration was increased to 3 wt.%, the Al-Fe intermetallic phases mostly appeared as long needles. For both concentrations, the weight percent of each phase corresponded to Al_6Fe but the concentration of Al may be overestimated because of the contribution of the Al matrix during EDX spot analysis. Previous researchers have identified these

Al-Fe intermetallic phases as Al_3Fe [15] or $Al_{13}Fe_4$ [16]. In the Al-3 wt.% Fe alloy, the Fe intermetallic are likely primary phases as the added Fe concentration is beyond the eutectic composition of 1.8 wt.% [17].



Location	Al (wt.%)	Fe (wt.%)	Ni (wt.%)	Si (wt.%)
A	96.4	3.6	-	-
B	82.7	16.6	-	0.7
C	72.6	27.4	-	-
D	86.6	13.4	-	-

Figure 2: SEM Images of a) Al-1.5 wt.% Fe and b) Al-3 wt.% Fe with Corresponding EDX Spot Analysis Results of Intermetallic Phases

Microhardness and Electrical Conductivity

The average electrical conductivity and average microhardness values of the prepared alloys are shown in Figure 3. The error bars represent one standard deviation above and below the average. The straight lines indicate lines of best fit for the conductivity and microhardness data. The average electrical conductivity for the pure Al condition was measured as 62.7% IACS and is indicated by the alloy composition of 0 wt.%. With increasing additions of either Ni or Fe, the electrical conductivity of the alloy continually decreased according to a linear trend, as expected. The addition of Ni tended to reduce electrical conductivity to a lower degree on a per weight basis as compared to Fe addition. The addition of Ni showed nearly a constant electrical conductivity of ~60 %IACS up to and including 1.5 wt.% and then dropped to 57 %IACS with 3 wt.% addition. The addition of Fe showed a gradual decrease in %IACS from 59 %IACS (with 0.5 wt.% addition) to 52.5 %IACS (with 3 wt.% addition). The Al-Fe and Al-Ni alloys still featured relatively high conductivity (>55 %IACS) with additions up to 1.5 wt.% of either element. Most cast Al alloys show a reasonable amount of Fe content (0.4-1 wt.%) to prevent mold sticking and reduce alloy costs. Therefore, an Al-Fe based alloy with even 1 wt.% Fe content would still have relatively high conductivity. In contrast, the microhardness of the alloys continually increased with the addition of Fe or Ni. The average hardness of the pure Al was measured as 25 HV, and the Al-3 wt.% Ni and Al-3 wt.% Fe alloys both showed hardness values near 37 HV. For addition levels up to and including 1.5 wt.%, the addition of Ni provided a greater hardness increase on a per weight basis than Fe. On a per weight basis, the observed changes in hardness and conductivity with the addition of Ni were +4.3 HV/wt.% and -1.7% IACS/wt.%, respectively. For Fe additions, the observed changes in hardness and conductivity were +3.9 HV/wt.% and -3.4% IACS/wt.%, respectively.

The hardness changes for the Al-Fe alloys on a per weight basis are similar to those of previous researchers of +4.16 HV/wt.% Fe, [5] but the reduction in conductivity (-1.4% IACS/wt.% Fe) was more severe in this study compared to previous research [5]. The differences may be due to previous researchers examining addition levels only up to 1.5 wt.% while this study examined Fe concentrations up to 3 wt.%.

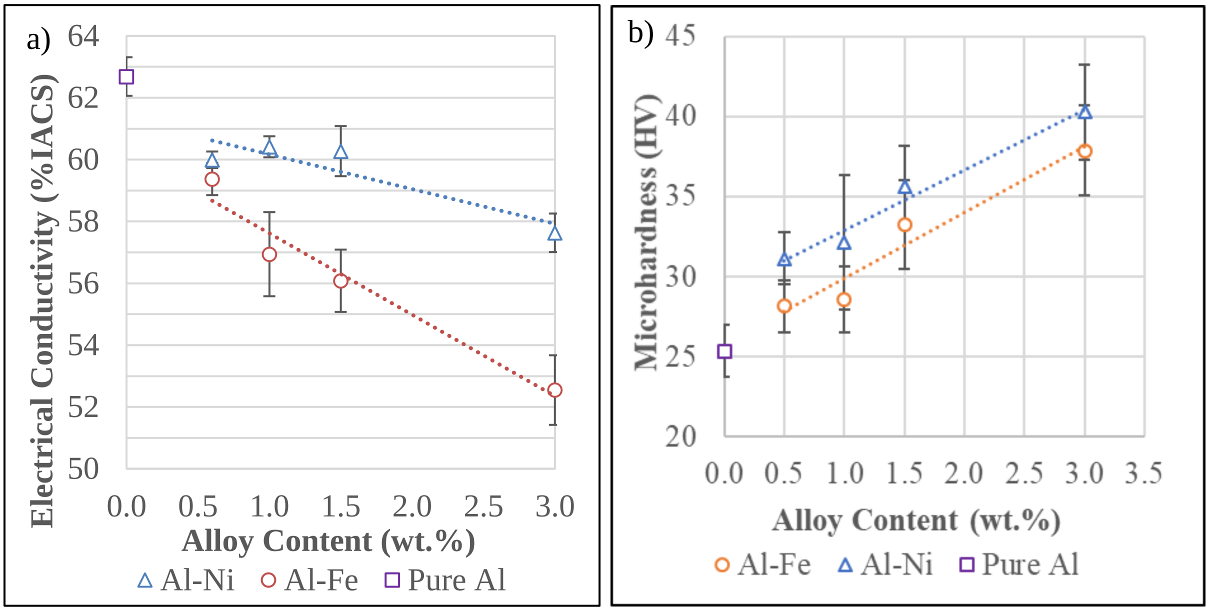


Figure 3: a) Electrical Conductivity and b) Microhardness of Pure Al, Al-Fe and Al-Ni Alloys

<u>Thermal Conductivity</u>

For aluminum alloys, electrical conductivity can be related to thermal conductivity using the modified Wiedemann-Franz Law [13] as shown in Equation 1.

$$\lambda = LT\sigma + c \qquad (1)$$

In Equation 1, k denotes thermal conductivity, L the Lorentz number (2.1 x10^{-8} WΩ/K^2), T is temperature in Kelvin (298 K) and c is the lattice thermal conductivity valued between 10.5-12.6 W/m-K [16,18,19] typically used for Al and Al-Si alloys. Estimated thermal conductivity values using the Wiedemann-Franz Law (Equation 1) from electrical conductivity measurements have shown very good agreement with measured thermal conductivity values by previous researchers [16]. The estimated thermal conductivity of the alloys are shown in Figure 4. A value of c=10.5 W/m-K was used to estimate a minimum thermal conductivity threshold while c=12.6 W/m-K was used to determine a maximum threshold. The electrical conductivity pure copper used was 5.8 x10^7 (1/Ωm) [17]. The thermal conductivities of the Al-Fe and Al-Ni alloys were above 200 W/m-K, even with addition levels of 3 wt.%. With increasing addition of Fe and Ni, more intermetallic phases were present which simultaneously increased hardness but also reduced TEC.

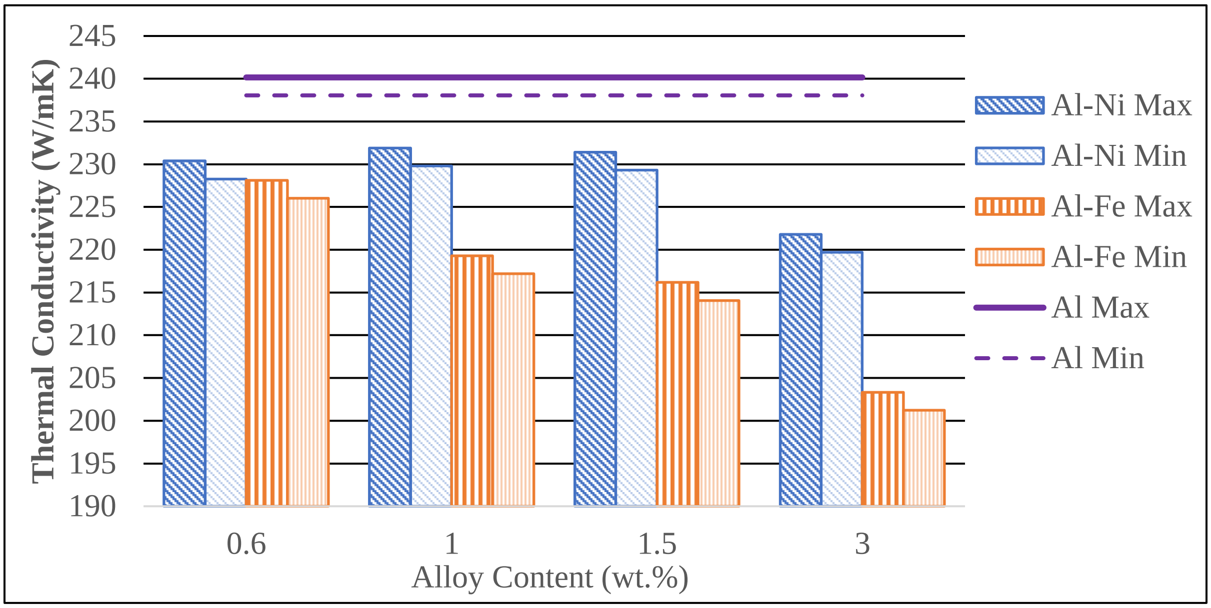


Figure 4: Thermal Conductivity of Pure Al, Al-Fe and Al-Ni Alloys

Conclusions

The effects of Ni and Fe additions on the TEC, microhardness and microstructure of pure Al were investigated. The major results were as follows:

1) In the 0.6-3 wt.% range, Ni additions reduced the electrical conductivity of pure Al by 1.7% IACS/wt.%, whereas Fe reduced electrical conductivity by 3.4% IACS/wt.%.
2) The calculated thermal conductivity of pure Al also reduced with Ni or Fe additions in the 0.6-3 wt.% range. The Al-3 wt.% Ni and Al-3 wt.% Fe alloys both had thermal conductivities >200 W/m-K, as determined using the Wiedemann-Franz Law estimation.
3) In the studied addition range, the microhardness of pure Al increased by 3.9 HV/wt.% for Fe addition. The increase in microhardness was due to the presence Al-Fe intermetallic phases. With Ni addition, the microhardness increased by 4.3 HV/wt.% due to fibrous and blocky Al_3Ni particles.

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