Solidification analysis of Al-Si alloy using in-situ neutron diffraction

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SOLIDIFICATION ANALYSIS OF Al-Si ALLOY USING IN-SITU NEUTRON DIFFRACTION

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ABSTRACT

In-situ neutron diffraction and thermal analysis techniques were used to evaluate the kinetics of the non-equilibrium solidification process of a hypereutectic Al-Si alloy using NRU nuclear reactor in Chalk River, ON and HFIR reactor in Oak Ridge National Lab, TN. Neutron diffraction patterns were collected in the stepwise mode during solidification between 740 and 400°C. The variation of intensity of the diffraction peaks was analysed and compared with the results of a conventional cooling curve analysis. It was shown that neutron diffraction offers potential for high resolution liquid and semi-solid microstructure analysis and can be used for novel studies on grain refining, eutectic modification, etc.

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INTRODUCTION

The hypereutectic Al-Si alloy (about 16-23% Si) is recognized as an excellent material for high performance automotive cast component applications. Important examples include linerless engine blocks for passenger vehicles, and motorcycle cylinder blocks cast using high pressure die casting (HPDC) and low pressure permanent mold (LPPM) casting processes [1-6]. Such components could be heat treated using T5 or T6/T7 tempers for better in-service performance. The hypereutectic Al-Si melts at near-liquidus temperatures are recognized to have a heterogeneous distribution of Si atoms (clusters) with a short range atomic order [3, 7, 8]. Most of these heterogeneities act as solidification sites and facilitate the primary Si nucleation [7]. Most likely they exist in the liquid state significantly above the liquidus temperature. For this reason, the as-cast microstructure is highly dependent on liquid alloy processing parameters such as melt and pouring temperatures, as well as solidification rate. These parameters have to be precisely controlled during industrial practice, to achieve castings with controlled size and distribution of primary Si crystals. Due to the complex solidification process, including the tendency for a heterogeneous distribution of alloying elements at near-liquidus temperature, hypereutectic Al-19%Si binary alloy was selected for advanced assessment using neutron diffraction and thermal analysis techniques.

Thermal analysis techniques are conventionally used for evaluation of the solidification process under semi-equilibrium conditions [3-5, 10]. Recent advancements in hardware and software development [9] allow for better control of solidification parameters, including a wide range of solidification and melting rates, as well as melt environment control (vacuum, inert/active, elevated pressure). The direct effect of a minor addition of alloying elements (parts-per-million levels) could be difficult to quantify using cooling curve thermal analysis. The search for improved phase identification and detection capabilities has resulted in an attempt to utilize the neutron diffraction technique for solidification analysis. The advantage of using neutrons lies in the highly penetrating nature of neutron radiation, which ensures that the scattering observed is representative of the bulk of a test sample. Neutron diffraction could provide direct and independent phase assessment, as well as quantification of the solid-to-liquid fraction during the alloy solidification process. The feasibility of this concept has been evaluated.

The objective of this paper is to assess the suitability of neutron diffraction techniques for advanced solidification analysis of hypereutectic Al-19%Si binary alloy.

EXPERIMENTAL PROCEDURE

Alloy Chemical Composition and Test Sample Configuration

The hypereutectic Al-19%Si alloy was used in the present studies. The binary-alloy chemistry (Table 1) was selected, to minimize the effect of other alloying elements on neutron diffraction and thermal analysis signals. Such an approach allows for better validation of the suitability of neutron diffraction techniques for solidification analysis. Once the system's sensitivity is established, more complex alloy chemistries will be evaluated. Typically, commercial hypereutectic alloy, such as 390, besides containing 16-19% Si, also contains additions of Cu up to 4%, and Mg up to 1%. A phosphorus addition of up to 0.1% is used for primary Si refinement. This alloy, as well as its modified versions, is used for a variety of high performance castings; e.g., engine blocks and pistons manufactured using HPDC and LPPM technologies [1-6].

Table 1 - Chemical composition of the investigated binary hypereutectic Al-Si alloy (wt%)

Si	Cu	Mg	Fe	Mn
19.3	0.013	0.01	0.1	0.01

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Thermal Analysis and Neutron Diffraction during the Alloy Solidification Process

Thermal analysis during the melting and solidification cycles was carried out using the UMSA Technology Platform [9]. The experiments were performed using cylindrical-shaped test samples with outer diameter (OD) = 16 mm and length (L) = 18 mm, and were heated to 785±0.2°C and isothermally kept at this temperature for a period of ten minutes, to stabilize the melt temperature and homogeneity. Next, the test samples were solidified at about a 0.5°C/s cooling rate, calculated between the non-equilibrium liquidus (start of the solidification process) and the non-equilibrium solidus (end of solidification process) temperatures, 672 and 543°C, respectively. To analyze the phase transformation temperatures for binary Al-19%Si alloy, the first-derivative-vs.-temperature and the fraction solid (FS) curves were automatically calculated and plotted. The detailed methodology is described elsewhere [3, 4].

A novel solidification cell was designed and built to carry out controlled melting and solidification experiments under the simultaneous exposure to neutron radiation. The crucible containing the analysed alloy is placed inside the open titanium tubing filled with Argon, to prevent oxidation at higher temperatures. The thermocouples were calibrated based on the melting temperature of high-purity Al, with a precision of ±0.5°C. The thermocouple insertion depth into the melt was limited to 6.5 mm, to prevent its contribution to the diffraction pattern. The crucible inner diameter was 18 mm, the total height was 63.5 mm (sample material height 57 mm), the sample volume was 14 cm^3, and its weight was about 32 grams. The neutron beam height in this setup was limited to 50 mm, to prevent "stray" peaks in the diffraction pattern coming from the control thermocouple. The test sample temperature was computer controlled based on the K-type sensor and PID circuit. The solidifying Al-19%Si test sample, within a temperature range of 740 to 400°C, was irradiated with monochromatic thermal neutrons of wavelength 0.237 nm using a Si monocrystal, and a {311} reflection. Diffraction patterns were collected isothermally at the following temperatures: 740, 695, 660, 630, 600, 570, 560, and 400°C. Experiments were performed at the Canadian Neutron Beam Centre, and measurements were taken in terms of scattered intensity vs. diffraction angle PHI=2 Θ (where Θ is the Bragg angle), using the C2 spectrometer of AECL's NRU reactor. The scattering angle ranged from 37 to 117 degrees for each run at each temperature.

EXPERIMENTAL RESULTS AND DISCUSSION

Thermal Analysis during the Solidification Process

Thermal analysis of the Al-19%Si binary alloy solidified at about a 0.5°C/s cooling rate (instantaneous 2.5°C/s in the liquid state) revealed the liquidus temperature to be at 672.4°C (see Figure 1 and Table 2). At this temperature, the first primary Si crystals nucleated from the melt. Evolved latent heat caused the temperature of the surrounding melt to rise. This point was clearly visible as a sudden change occurring in the first derivative curve (point #1 in Figure 1b). As the solidification process continued to progress the primary Si crystals continued to grow. At 568.9°C, the next abrupt change in the first derivative curve corresponded with the nucleation of the Al-Si eutectic (point #2 in Figure 1b). This was manifested by the positive value of the first derivative peak (see Figure 1b). Solidification was completed at 542.7°C, which corresponded to the solidus temperature (point #4, Figure 1b). The FS vs. temperature curve for a solidification rate of 0.5°C/s is presented in Figure 1c. A temperature of 672.4°C signifies the nucleation of the primary Si crystals and corresponds to 0% FS. The growth of FS of the primary Si crystals as a function of temperature is linear up to 20.5%, which corresponds to the temperature of 568.9°C at which the Al-Si eutectic starts to nucleate.

The approximate 20% FS at this point can be considered to be the volume fraction of primary Si, since this is the only phase that nucleates from the liquid within the temperature range of 672.4 - 568.9°C (see Figure 1c, Table 2). The temperature interval for the development of the primary Si crystals was very wide - about 103°C. In contrast, the nucleation and growth of the Al-Si eutectic was linear and took place under an almost constant temperature of about 569°C. For example, at 50% progression of the Al-Si eutectic reaction, the overall FS in the investigated alloy was about 64% (including both primary and eutectic Si) (see Table 2).

Table 2 - Non-Equilibrium Thermal Characteristics of the Al-19%Si Binary Alloy Obtained during the Solidification Process

No	Thermal Characteristics	Temperature, °C ±STDEV	Fraction Solid, %
#1	Nucleation of the primary Si (liquidus temperature)	672.4±0.6	0
#2	Nucleation of the Al-Si eutectic (start)	568.9±1.5	20.5
#3	Nucleation of the Al-Si eutectic (50% progression)		64
#4	End of solidification (solidus temperature)	542.7±1.3	100
	Solidification range	129.7	

Metallographic observations of the binary Al-19%Si test sample that solidified under an average cooling rate of 0.5°C/s revealed coarse, unrefined primary Si and unmodified Al-Si eutectic (see Figure 2). The length of the primary Si crystals was found to be up to 500 µm. It was observed that the microstructure of such a slowly solidified thermal analysis test sample was highly heterogeneous, caused by the lack of primary Si refinement and the tendency of primary Si for gravity segregation due to its lower density.

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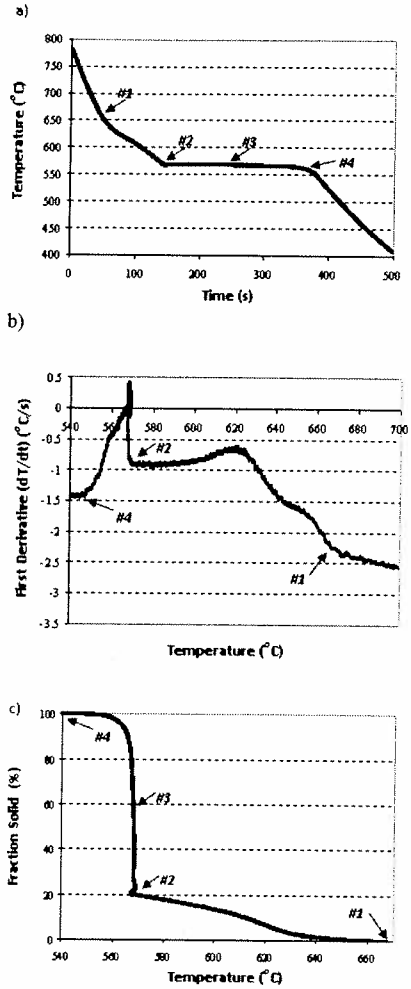

Figure 1 – Cooling curve analysis results obtained for Al-19%Si binary alloy solidified at an average cooling rate of about 0.5°C/s a) Temperature vs time curve b) First derivative (dT/dt) vs temperature curve c) Fraction solid vs temperature curve

Note: The corresponding microstructure is presented in Fig. 2 the numbered arrows correspond to the metallurgical reactions and are outlined in Table 2

Neutron Diffraction during the Solidification Process

The neutron diffraction experiment was started with a no-sample furnace set-up, to retrieve the "background" that will be present in all measurements. For the selected neutron wavelength of 2.37 Å, several diffraction peaks observed in the "background" pattern can be easily attributed to various crystallographic planes reflections of titanium and steel - the materials used to build the solidification cell. Simple subtraction of the background data from the original raw-data patterns collected for the set-up with the sample metal typically results in a clearer "sample-only" diffraction pattern. Figure 3 depicts diffraction patterns received throughout the experiments, as the Al-19%Si melt temperature was reduced in

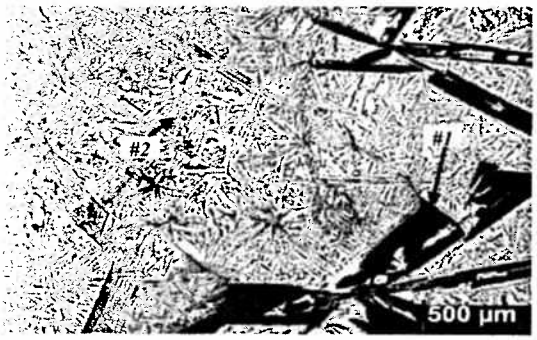

Figure 2 - Optical micrographs (50x) of the Al-19%Si binary alloy test sample solidified at an average cooling rate of about 0.5°C/s. Note unrefined primary Si crystals up to 500 µm long (#1) and unmodified Al-Si eutectic (#2)

the stepwise mode from 740 to 695, 660, 630, 600, 570, 560, and finally, to 400°C. As expected, no new peaks were observed in the pattern received for the molten alloy at temperatures above liquidus at 740°C, but the diffraction intensity through the entire range of the scattering angle was increased due to inelastic scattering in the melt. The curved shape of the 740°C-line (as compared, for example, to the 400°C-line) suggests that it represents a pattern received for a liquid sample [12]. Other diffraction patterns in Figure 3

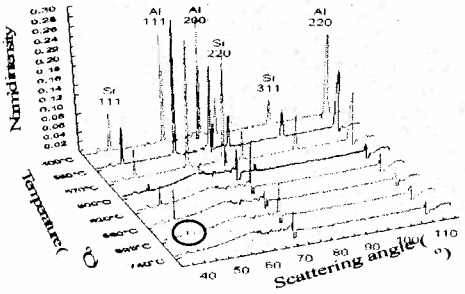

Figure 3 - Neutron diffraction pattern of the Al-19%Si binary alloy solidification process collected at various temperatures, ranging from 740°C (45°C above the equilibrium liquidus temperature) to 400°C (143°C below the solidus temperature)

show peaks that represent diffraction by several crystallographic planes of the solid Al and/or Si crystals through the solidification interval. Integrated intensity of the peaks received at 400°C represents 100% FS.

The apparent peaks that could still be observed in the vicinity of the 70° and 110° scattering angles for the overheated molten Al-19%Si binary alloy was an artefact of a small temperature mismatch between the background data set and the sample data. These apparent peaks overlap with two crystallographic reflections of the sample material - namely, [200] and [220] reflections of Al. Unfortunately, this interference caused too much of an error for the [200] reflection, so this plane had to be removed from further analysis. As for the other diffraction peaks, as follows from Figure 3, the integral intensity can be used as reliable information that directly reflects the relative amount of solid phase that contributed to the neutron scattering. Analysis of the diffraction pattern for the Al-19%Si melt at 695°C showed that some solid Si exists in the melt. The diffraction peak (circled in Figure 3) that exists at about 44 degrees can only be attributed to the [111] reflection of Si. It is to be noted that 695°C is about 23°C higher than the liquidus temperature established from the thermal analysis experiments. Based on the intensity of this diffraction peak relative to the peak intensity at 400°C, the FS of Si in the melt at the given temperature will be quantified later in this paper. Correspondingly, by collecting the diffraction patterns for the semisolid alloy for various temperatures within the solidification interval, one can retrieve valuable information on the dynamics of non-equilibrium solidification; for example, FS of Al and FS of Si vs. temperature of the melt,

as a result of direct measurement and calculation of the integrated intensity of the diffraction peaks. This will also be discussed further in this paper.

Analysis of Fraction Liquid using Neutron Diffraction

Direct fitting of Gaussian functions into the diffraction peaks during solid-phase analysis could ignore the sample background underlying these peaks, which contains very useful information. It was reported in the literature [12] that there is decrease in the background intensity underlying the solid phase peaks. A correction was required to account for the drop in the level underneath the peaks, which would cause a systematic understatement of the solid phase content as a function of temperature. This also implies that, if care is taken, then neutron diffraction is capable of simultaneously being an independent monitor of the proportions of both liquid melt and solid alloy inside the solidifying test sample. Figure 4 represents the diffraction patterns received for the sample material at different temperatures. There is a clear difference in the intensity of the background signal for all between-the-peaks areas, which originates from the reduction in fraction liquid in a course of alloy solidification. This means that it is relatively simple to separate melt and solid scattering. This separation is done by performing a simple integration of scattering intensity over the range in which the change in the scattering pattern is only due to the change in the proportion of liquid [1, 11]. The regions of the diffraction pattern immediately beyond the solid-alloy peaks can therefore be used as an indicator of liquid content in the sample volume. Three such regions were identified in Figure 4; namely, 45 - 59 degrees, 77 - 91 degrees, and 93.5 - 108 degrees of the scattering angle. Normalization was done in such a way that the initial liquid content at 740°C was 100%, and the liquid content at 400°C was 0%.

The results of this analysis are shown in Figure 5, along with two lines that represent the non-equilibrium solidus and liquidus temperatures determined from the thermal analysis carried out during the Al-19%Si binary alloy solidification process (see Figure 1 and Table 2).

It can be seen that all three curves obtained from the neutron data analysis are in reasonably good agreement. The fraction liquid is about 95% at about 10°C below the non-equilibrium liquidus temperature, and most of the solid phase evolves at the later stage of solidification. It can be concluded from Figure 5

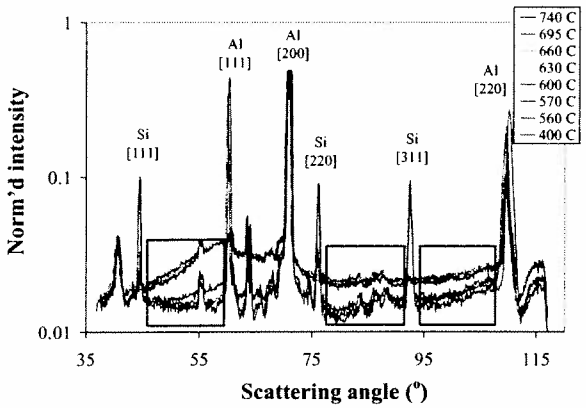

Figure 4 - Neutron diffraction pattern collected during the Al-19%Si binary alloy solidification process. Selected no-peak areas were used for the fraction liquid analysis

and Figure 1c that most of eutectic phase evolves between about 569°C and 560°C, and down to 543°C. We note, however, that we observed a diffraction peak from solid Si at 695°C (about 23°C above the non-equilibrium liquidus determined based on the thermal analysis experiments), see Figure 3. This peak indicated the presence of primary silicon crystals in the liquid alloy. As it is impossible to quantify the FS (of Si) using the liquid-phase data, we will try quantifying it while evaluating the scattering intensity by the solid-phase crystals.

Analysis of Fraction Solid using Neutron Diffraction

Figures 6 (*a*) and (*b*) depict the Si [111] and Al [220] peak evolution over the solidification interval and further cooling to 400°C. The integral intensity of the diffraction peaks is clearly increasing as

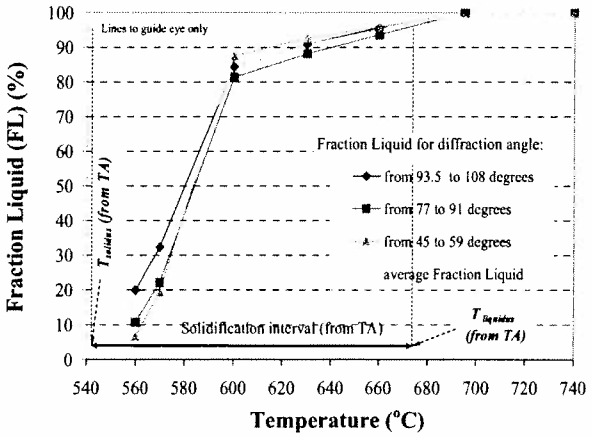

Figure 5 - Fraction liquid curve obtained from neutron diffraction analysis of the Al-19%Si binary alloy solidification process

each peak becomes higher and wider. The angular position of the peaks also changes as the temperature decreases, which corresponds to thermal contraction of the solid metal (i.e., a reduction in the lattice plane spacing reflected by the shift in Bragg's peak position). Similar to the liquid phase analysis, by performing a simple integration of the peak's normalized intensity over the angular range that covers the width of the peak, one can retrieve the relative FS for the selected temperatures within the solidification interval of the alloy. The basic approach taken in this study was to simply prorate the integral intensity from zero at 0% solid (above $T_{liquidus}$) to 1 at 100% solid (below $T_{solidus}$). The results of separate calculations performed for Si [111] and Al [220] are presented in Figure 7. In addition to this, Figure 7 also contains a fraction-solid profile received from the bulk liquid analysis as FS = 1 – FL. The non-equilibrium solidification range and FS vs. temperature solidification profile determined by thermal analysis are also marked on the graph (see Figure 7). An important observation from Figure 7 is that solid Si starts evolving in the melt much sooner than solid Al, which is in agreement with the solidification sequence for the studied alloy. The presence of Si crystals (i.e., clusters) can be detected above the liquidus temperature (FS_{Si} about 3%) and rapidly evolves to be in excess of 20% about 12 degrees below the liquidus temperature. On the other hand, solid Al evolves very slowly as the metal temperature decreases, but accelerates rapidly at temperatures below 600°C. This observation corresponds well with the earlier results of thermal analysis (see Figure 1) and liquid phase analysis (see Figure 5), which indicated that most of the alloy solidifies as eutectic within the temperature range of 570–560°C (to 90~95% solid), with solidification completion at about 543°C. The "overall" solidification profile represented by the line that was derived from the liquid-phase analysis matches well with the results received for the solid Si and solid Al evolution, though, obviously, the profile lacks Al/Si-related specifics. The same is also true for the other line that also represents the "overall" solidification profile based on the thermal analysis data.

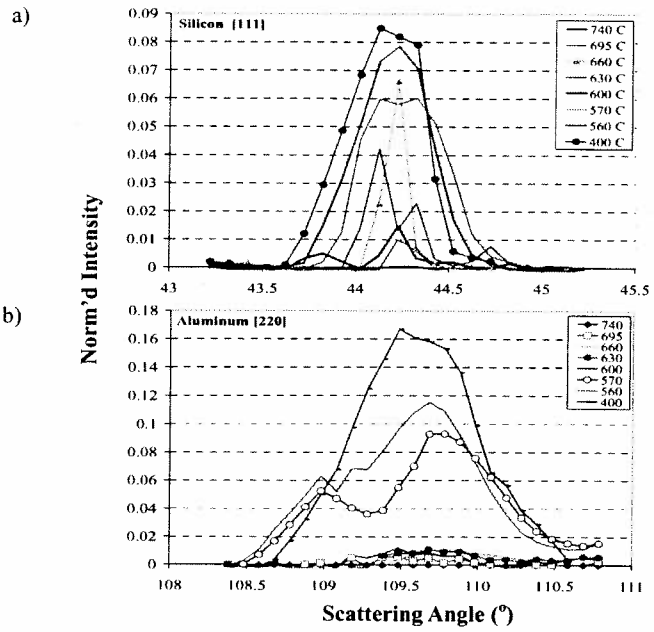

Figure 6 - Diffraction intensity change in the neutron diffraction experiments recorded during the Al-19%Si binary alloy solidification process between 740 and 400°C, for the following phases: a) Silicon [111], b) Aluminum [220]

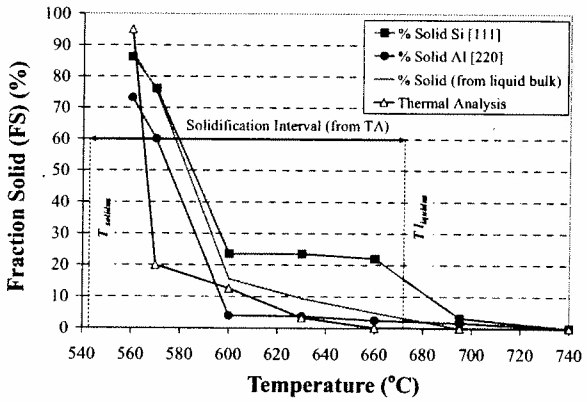

Figure 7 - Fraction solid evolution obtained during neutron diffraction analysis of the Al-19%Si binary alloy solidification process. Data were received from "liquid bulk" analysis and derived from Si [111] and Al [220] diffraction peaks

Regardless of the "classical" use of neutron diffraction for studies of solid metals in metallurgical research, the results presented show the potential of applying neutron diffraction for high resolution analysis of liquid and semi-solid alloy. It will be particularly interesting to explore further the near-liquidus melt characteristics, since neutron data revealed a Si signal above the liquidus temperature established by thermal analysis. The clustering of Si atoms prior to the start of the solidification process is not fully understood and requires further clarification, since it significantly affects primary Si size and distribution in cast components. This understanding can result in new characterization capabilities for detailed studies

of grain refining, eutectic modification, etc. This outcome will contribute to better understanding of the solidification behaviour of the Al-Si hypereutectic alloys during casting processes such as HPDC and LPPM.

CONCLUSIONS

Neutron diffraction was used for the first time in studies of non-equilibrium solidification of Al alloys. This study focused on hypereutectic, binary Al-19%Si alloy. The neutron data collected allowed for no-bias analysis of the solidification process. These findings were supported by results of thermal analysis, as well as by microstructure evaluation. The following characteristics of non-equilibrium solidification of the Al-19%Si binary alloy were revealed in this research:

1. Thermal analysis performed on the solidifying alloy revealed a solidification range of 129°C.
2. Thermal analysis and microstructural evaluation also revealed that the growth of FS as a function of temperature is linear up to 20.5%. This FS corresponds to a temperature of about 569°C, at which the Al-Si eutectic starts to nucleate. Most of the solid phase evolves as eutectic at the last stage of solidification within a very short temperature range below about 569°C.
3. Neutron diffraction indicated that there is solid Si present above the non-equilibrium liquidus temperature of 672°C. The content of solid Si in the melt at 695°C exceeds 3%. Si phase evolution above the liquidus temperature can indicate the agglomeration of clusters of primary Si.
4. Neutron diffraction could clearly detect the presence of solid Al only at 660°C, well below the liquidus temperature.
5. Neutron diffraction data confirmed that there is an obvious difference in the solidification path of Al and Si, with the latter evolving at a much higher rate during the early stage of solidification of the alloy (primary Si).
6. It was confirmed that the intensity of neutron scattering by the liquid metal can be used as an independent indicator of fraction liquid in the melt.
7. Accelerated solidification that can be observed in the solid-phase neutron diffraction experiments between 570°C and about 560°C matched well with the findings of the liquid-phase neutron diffraction analysis and the thermal analysis of the solidification process. This confirms that most of the FS evolves as eutectic within the temperature range of 569 to about 560°C, and down to 543°C.

Based on the above results, further development of methods to apply neutron diffraction for studies of the non-equilibrium solidification of metal alloys is well warranted.

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