

## Microstructure Control by Solidification Processing—Outcome from a European Thematic Network\*\*

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The MEBS network gathered together 20 institutions, comprising 8 industries, 6 research centers and 6 universities, for a four year project (1999–2002). It was organized in four subgroups with a full meeting each year to which speakers from outside of the network were also invited. Moreover, a few non-funded institutions have been also active partners. On the whole, more than 70 persons have attended at least one of the yearly meetings. The first idea for this network arose long ago during a meeting of an informal group of senior scientists working in the field of solidification in Europe. Among the many subjects which were mentioned to reinforce ties between academia and industry as well as between laboratories in Europe, four topics were finally selected with well-defined work programs. Networking was also intended to promote dissemination of scientific information and this special issue of the AEM journal was designed to fulfil part of this duty. Some aspects addressed during the implementation phase of the network do not appear in this series of papers as

they have been published or will appear in other journals. All the work under the network is, however, outlined in this foreword, taking each topic in turn.

Topic 1: Nucleation and grain refinement (coordinator: A. L. Greer; alg13@cam.AC.uk).

Nucleation of crystalline grains is relevant in all of solidification processing, but studies of nucleation are most active for aluminium and its alloys. For these, inoculation by addition of nucleant particles to the melt is the usual practice to ensure a fine equiaxed grain structure. Research on this grain refinement process has been particularly active over the past few years as the aluminium industry faces the general challenge of cost reduction and more specific issues such as the optimum treatment of recycled metal. Subgroup one of the MEBS network brought together industries and universities with active research programmes on grain refinement; the core partners were: Alcan International Ltd (UK), University of Cambridge (UK), London & Scandinavian Metallurgical Co. Ltd (UK), University of Oxford (UK), Pechiney (F), University of Swansea (UK), and VAW (D). Under the network, links were also established with further industries and research groups in Finland, France, Germany, the Netherlands, Norway, Spain, Switzerland, and the UK, and outside Europe with a group in Australia. The interests of the participants covered shaped casting and direct-chill (DC) casting of wrought alloys. Importantly, the industries in the network included both a manufacturer and users of grain refiners. For Topic 1, the role of the MEBS network has been mainly to facilitate exchange of information and coordination of research activities. New links have been established, especially between academic and industrial partners and many joint publications<sup>[1–47]</sup> as well as internal reports have resulted. The review article in this issue summarizes the work on Topic 1; key aspects of this are briefly outlined here.

The most widely used refiners are based on the system Al–Ti–B. While these can be very effective in suppressing columnar growth and in giving fine equiaxed grains, they do have problems. These include a tendency to agglomerate giving undesirably large particulates in the cast product and a susceptibility to poisoning, i.e., reduction or destruction of their effectiveness in the presence of certain solutes in the melt. For these reasons there has recently been much interest in the use of Al–Ti–C refiners which do not agglomerate and which appear resistant to some types of poisoning. For the MEBS network a central theme was the comparison of Al–Ti–B and Al–Ti–C refiners.

Improved fundamental understanding of nucleation mechanisms was achieved by transmission electron microscopy (TEM) which shows that the TiB<sub>2</sub> particles in Al–Ti–B refiners may not be stoichiometric, with important implications for interpreting the performance of stoichiometric refiners (i.e., those without Ti in excess of that bound in boride particles). Analysis of the mechanisms of growth-restriction and modeling of the effect for multicomponent systems of industrial relevance were completed. Development of a free-

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growth model has allowed quantitative prediction of grain size as a function of refiner addition level, cooling rate and solute content. Further analysis of how additions of grain refiner affect the selection of secondary intermetallic phases in wrought alloys has also been considered. TEM shows also the different mechanisms by which Al-Ti-B refiners are poisoned by zirconium and by silicon.

Assessment of the new Al-Ti-C refiners involved microstructural verification that the nucleant particles are TiC and that the largest of these are active in grain initiation. The grain-refining performance has been compared with Al-Ti-B refiners. Calculation of the Al-Ti-C phase diagram showed that the TiC particles are unstable in inoculated melts, but it was demonstrated that the dissolution of TiC is sufficiently slow not to impair refining performance. Silicon poisoning was characterized and its mechanism elucidated (transformation of TiC to SiC). The free-growth model appears to work also for these refiners and it helps to interpret the differences in performance between Al-Ti-C and Al-Ti-B refiners.

From these results, it appeared possible to define the best refining practice for conventional alloys and refiners. This was checked by means of extensive tests of a variety of refiners for DC casting, roll casting and shaped casting, but also tests of the effects of refiners on filtration. Use of quantitative modeling to interpret refiner performance and to facilitate the optimal choice of refiner type and addition level was carried out. In the same line of study, the possible improvements in refiner performance were explored by extensive calculations on the effects of nucleant particle size distribution on refiner performance and study of the particle size distribution in existing commercial refiners. Improvements in performance of the refiners would be possible by having a narrower spread in particle size, thinner nucleant particles, and by choosing an average particle size most appropriate for the particular cooling rate and solute content. Finally, a nucleation law for application in solidification modeling was established. The undercooling for grain initiation is given by the free-growth model and has been applied in cellular-automaton finite-element modeling of the columnar-to-equiaxed transition.

The major deliverable on Topic 1 will be a book summarizing the state of the art in grain refinement. The book "Grain refinement of Alloys by Inoculation of the Melt: Theory and Application to Al-based Alloys and other Systems" is at present in advanced stages of preparation. It has 19 chapters, written by 13 authors, of whom 10 are from core partners in the MEBSP network. Seven MEBSP partner institutions are involved, four industries and three universities.

Topic 2: Solidification and microsegregation (coordinator: A. Howe; andy.howe@corusgroup.com). The network on this topic comprised representatives from Corus (UK), CIRIMAT (France), DLR (Germany), IRSID (France), IST (Portugal), KTH (Sweden), Siemens-PG (Germany), RWTH (Germany), and VTT (Finland), with interests in steel, aluminium, and nickel-base alloys. The objectives were to compare experimental methodologies, to characterize solidification kinetics

and microsegregation, and to compare numerical tools for solidification kinetics and the associated build-up of microsegregation. In both cases, the aim was for the work and cross comparison to lead to developments in experimental and simulation techniques and interpretation. The main aspects of the work satisfying these objectives are now briefly described in this overview, with further detail in accompanying papers.

Some issues concerning experiments and models for alloy microsegregation were analyzed. A "catalogue" of various considerations, problems, and unusual features for microsegregation studies has been prepared, and constitutes a paper in this special issue. For simulations, the common assumption of local equilibrium at the interfaces generally seems to be satisfactory, but in many cases there is still considerable uncertainty in the accuracy of the equilibrium data. There is generally much more uncertainty regarding the solute diffusivities, to the extent that there is often little point in seeking great accuracy for other factors such as the dendrite scale and the arm coarsening behavior. The importance of the thermal conditions is emphasized. Some experiments or model simulations employ a constant cooling rate, which is a very unusual thermal history in industrial practice, and can have a major effect on the kinetics and indeed upon the dendrite morphology. Some basic checks on experimental analyses are recommended and consideration is given to what data are recorded and how they are presented. In particular, the advantages and disadvantages of specific line-scans and integrated/cumulative profiles are discussed. The latter often gives a "tail" to the distribution at low concentrations which is not present in line-scans, resulting from a combination of random scatter in the individual analyses and the presence of various length-scales and their associated severity of segregation profiles, in the area integrated. However, a classic peritectic reaction can produce such a tail (or peak, depending on the element) at the dendrite cores, as found on line-scans. The determination of fraction solid is notoriously misleading from quenched-out samples. Substantial amounts of solid are deposited during the quench on the existing, coarse morphology, before the system responds to produce a fine scale, "quenched" structure. Finally, a range of unusual segregation features is described.

Direct comparison of microsegregation simulations were performed. The in-house Corus model for microsegregation<sup>[48]</sup> was compared in a mini "round robin" with those from the Technical University of Darmstadt and Ecole Polytechnique Fédérale de Lausanne within a recent COST Action,<sup>[49]</sup> for single phase solidification of a binary in planar, cylindrical and spherical fashion, and for a binary exhibiting secondary dendrite arm coarsening according to power laws. The comparison employed the same input data regarding geometry and thermophysical data, and the results were in encouragingly good agreement. The opportunity was taken within the MEBSP network to include comparison with a phase-field model from ACCESS eV,<sup>[50]</sup> and to extend this to

tracking a solid/solid interface during a thermal cycle, and to a multicomponent, peritectic case with arm coarsening and sub-solidus homogenization. The one-dimensional (1D) simulations again showed very good agreement, but in the 2D case, the inherently "blurred" interface of the phase-field approach was not compatible with the imposition of a fixed, curved cell boundary and the simulation ended with localized spots of segregate around the assumed, cylindrical form. The phase-field approach is much more successful when left to calculate the natural 2 or 3D morphologies for itself, rather than being forced to adopt particular ones. Regarding the multicomponent peritectic case (iron solvent with one rapid diffuser, carbon, and one slow diffuser, manganese) discrepancies were noted with the Corus model regarding the velocity of the solid/solid (body-centered cubic (bcc) ferrite/face-centered cubic (fcc) austenite) interface. These were found to be due to the approximation made in the Corus model of complete mixing of carbon in each phase. This approximation is very successful for either ferritic or austenitic solidification, but evidently not for the ferrite/austenite reaction itself, even though the carbon profiles are very nearly flat.

Study of kinetics and morphology of the solidification of binary and ternary aluminium alloys constitute also a paper in this special issue. The investigations in this network aimed to describe the microstructural features and the microsegregation in binary AlSi and ternary AlSiMg alloys when using the same solidification procedures. The special new point is not only to link microstructure and microsegregation, but also to measure in-situ during directional solidification with high spatial and temporal resolution the processing conditions necessary to ensure that the local solidification velocity and the local temperature gradient match as closely as possible. For directional solidification we used a new type of furnace named ARTEMIS (AeRogel TEchnology for Microgravity Solidification). It utilizes the extreme properties of nanostructured materials called aerogels. The experimental results are compared with numerical modeling for microsegregation and accepted steady-state growth models of solidification for the microstructural features. The observed differences between well-accepted theories and experimental results suggest that the theory of primary spacing or mush evolution needs more attention and refinement. Also the aspect of convective mass transport within the mush needs to be developed further and even the seemingly well understood eutectic solidification seems to need refinement with respect to growth in the confined geometry of the mush.

Solidification and heat treatment of Ni-base superalloys was also considered. Nickel-base superalloys are high-temperature engineering materials with an excellent high-temperature strength which mainly arises from the homogeneous precipitation of cuboidal  $\gamma'$ -precipitates in a Ni-rich fcc  $\gamma$ -matrix. The optimum microstructure is a uniform distribution of cuboidal small sized  $\gamma'$ -precipitates. However, solidification of Ni-base alloys gives rise to segregation, and as-cast materi-

al shows coarse residual  $\gamma$ - $\gamma'$ -eutectics and irregularly shaped  $\gamma'$ -precipitates formed during cooling. Thus, Ni-base superalloys generally require heat treatments including solutioning, homogenization and precipitation stages. Different heat treatments were performed and the specimens analyzed. Only a "full homogenization" treatment was actually successful, i.e., giving re-resolution of the original  $\gamma'$ -precipitates and considerable reduction of the extent of microsegregation, as the shorter versions led to poorer microstructures.

Topic 3: Thermophysical data (coordinator: Andreas Ludwig; Ludwig@gi.rwth-aachen.de). This subgroup comprised representatives from four companies, HYDRO (in the past VAW, Germany), PECHINEY (France), SIEMENS-PG (Germany), TEKSID (Italy), from six research organizations IKTS (Germany), INTOSPACE (Germany), IfG (Germany), NPL (UK), ÖGI (Austria), and VTT (Finland), and from the universities RWTH (Germany) and USTUTT (Germany). The main objectives of the cooperation within this subgroup were i) measurement and validation of data directly related to the partners and ii) compiling information on thermophysical data, methods, and measurements. The outcome of the cooperation within subgroup 3 is briefly described below while more detailed contributions can be found either in this issue or in the cited publications.

Numerical modeling of casting processes has become an up-to-date standard in industry, which aims at a short term design of modern casting with optimized properties. However, beside standard simulation techniques, there exist many sophisticated models with quite promising results and so it may be difficult for a non-expert to have an overview of the material properties necessary for such advanced casting simulations. A review outlining the present modeling approaches and introducing the thermophysical properties necessary for such simulations was produced.<sup>[51]</sup> Mould-filling and heat-transport simulations, stress and strain predictions, cellular automaton and phase-field techniques, and recent multiphase volume-average approaches were discussed. It was shown that the number of necessary material properties is directly correlated to the level of complexity considered in the corresponding modeling approach.

As casting simulations can be more or less detailed it is appropriate to classify the important material properties according to the complexity of the simulation.<sup>[52]</sup> Materials properties necessary for a simulation which considers only heat conductivity in the casting and the mould are defined as "basic". If mould filling is included in the numerical model further properties have to be known. As (even approximate) mould-filling simulations are standard nowadays, these properties are grouped in the "standard" class. Recently, more and more computer codes simulate residual stresses and distortions. However, due to different expansion/compression behavior of casting and mould material, gaps between the casting and the mould as well as areas of increased contact pressure may appear. These phenomena can greatly influence the heat extraction from the casting. Such simula-

tions are still at the development stage (see Topic 4). Therefore, we group the material properties needed for those simulations in the "advanced" class. This is also true for properties which are characteristic for the liquid/solid region like permeability of the mush or settling constants for moving grains. Those quantities are important for computer codes which model more specific solidification.<sup>[53]</sup>

The subgroup members have gathered their experience in searching and/or determining thermophysical property data relevant for casting simulations on a special web site which was presented at the Materials Week in Munich 2000.<sup>[52]</sup> It comprises information on i) reference books; ii) databases; iii) measuring techniques; iv) measurers; and v) theoretical/empirical models to predict properties. We have grouped information on reference books into two lists. The first list contains books where data are explicitly given and the second those on measuring techniques. When possible we have provided a link to corresponding information given by the publisher. Although not sufficient for finding certain properties of given materials, a short description of the contents of the corresponding book is given. Online access to the full table of contents would be useful, as with that it would immediately be obvious whether the corresponding reference book gives information on the important data or not. However, this was not possible to realize within the scope of the network. Existing databases were looked for on the WEB which showed different degrees of completeness. Some of them are available online free of charge. In order to gain an impression of the quality of the databases we looked at the following features: i) availability of temperature-dependent data, ii) commercial purpose, iii) possibility of online access, iv) data on solids, v) data on liquids, vi) available materials, and vii) available data on our benchmark alloy AlSi7Mg (see below). For each of the databases our web site gives a short description of its contents and, when possible, a list of the available properties. The direct link to the database supplier may give the possibility of further information.

For measurement of thermophysical properties of solid materials, a wide number of techniques do exist. As a start, we focused our survey on measuring techniques for properties of liquids. This includes measuring techniques for enthalpy and specific heat, thermal conductivity and thermal diffusivity, density and surface tension, viscosity and emissivity. In addition, the web site gives a short description of the advantages and the disadvantages as well as further characteristic details about each technique. This includes the common temperature range together with the materials which can be processed with that technique. When possible, measurers with experience in using the corresponding technique are designated. As the knowledge and the experience of the subgroup members is of course limited, the information given at the web site is not exhaustive and supplementary information as well as critical remarks are highly welcome. To further update the web site, NPL has agreed to pay attention to it beyond the end of the MEBS network.

Benchmark tests on measurements of thermophysical properties of selected Al-base alloys were performed with density, surface tension, viscosity, thermal conductivity, and electric resistance, all as function of temperature, as selected properties. Several ternary AlSiMg alloys and several commercial AlSiCu+ multicomponent alloys were selected. All in all, 320 samples with 21 different compositions were produced by HYDRO and distributed among the laboratories. Theoretical predictions with the virtual measuring system were based on the beta-version 0.08.1 of the NPL system and the NPL aluminium database v5.01 (VMAI). Calculations for Al-Si-Mg and Al-Si-Cu alloys assuming a "Scheil" solidification were performed to predict heat capacity, thermal conductivity, density and solid fraction. It turned out that additions of Mg and Cu significantly affect the solidification behavior and the thermal properties of Al-Si alloys. Also the properties of Al-7Si-0.3 Mg are changed significantly by adding 1 wt.-% Fe to the alloy. The electrical resistivity of some of the above mentioned Al-base alloys were measured up to and above the melting temperature by a new technique developed at the University of Stuttgart.<sup>[56]</sup>

As for the Al-base alloys, a selected single-grain Ni-base alloy was considered for a benchmark test of thermophysical properties.<sup>[55]</sup> For this purpose a great number of samples of the same batch were tested in six laboratories by using a variety of different measurement techniques for the following properties: linear thermal expansion coefficient, density, specific heat capacity, thermal diffusivity, thermal conductivity—all as function of temperature from room temperature to 1200 °C. Some properties were directly measured, others deduced from the measured ones. The results of the measurements were compared and derivations discussed. The overall result of this co-operation is that within the limits of measurement accuracy and up to around 1000 °C the estimated properties agreed reasonably well with each other. Above 1000 °C we found significant deviations which may be caused by dissolution of  $\gamma'$ -precipitates.

In order to improve the results of numerical process simulations, particularly with long solidification and cooling times, work has been carried out to establish more precise thermophysical data for mould materials and on the heat transfer coefficient between casting and sand mould.<sup>[57]</sup> IfG has carried out a research project to investigate and to establish the relevant thermophysical properties of resin-bonded and bentonite-bonded operational mould material and laboratory mixtures. As large castings, where long solidification and cooling times occur, are predominantly produced in no-bake sand moulds the results are mainly concerned with cold resin-bonded mould materials. Measurements of enthalpy, specific heat capacity, and thermal conductivity for this mould material has been performed as function of temperature. The thermal conductivity was measured with both a steady state and an inverse method. In addition, the heat-transfer coefficient between cast iron and the mould material was estimated.<sup>[57]</sup> The measured thermophysical mould mate-

rial data and the determined heat-transfer coefficients have been used in corresponding numerical simulations. The results of these simulations were then compared with those from casting tests and with simulations carried out with standard data sets. It was concluded that the use of the measured values of effective thermal conductivity, specific thermal capacity at elevated temperatures and heat-transfer coefficients improves the results of the simulation especially for the end of solidification. The use of standard data sets leads to solidification times which are too short.

Topic 4: Thermal stresses and strains during solidification (coordinator: Hasse Fredriksson; hassef@matpr.kth.se). Because most metals shrink during a cooling process, thermal stresses develop in the material if the cooling rate differs in different locations of a component. Thermal stresses are a very severe problem in casting processes where they cause hot cracks, and thus have a very large influence on the yield. Also, strains related to metal shrinkage may lead to the formation of an air gap between the mould and the cast component which in turn affects heat transfer. Thermal contraction causes other problems as well, such as macrosegregation. The cooperation in sub-group 4 has concentrated to study the mechanisms behind hot-crack formation and air-gap formation in permanent mould castings, both experimentally and by simulation. The main partners in this sub-group were Alcan (UK), CEMEF (France), KTH (Sweden), RWTH (Germany), and VTT (Finland).

The heat transport during a solidification process is strongly affected when an air gap develops. The relationship between variations in the heat-transfer coefficient and the formation of an air gap has been studied in mould casting processes. It has been shown that the gap formation starts as soon as the solid metal shell is strong enough to withstand the pressure from the liquid metal. Before the formation of a macroscopic air gap, heat transfer occurs mainly through conduction, while when the air gap starts to grow, the conduction gradually decreases and the heat transfer can be described by a simple superposition of the radiation and conduction terms. In order to study the solidification process coupled with the air gap formation an experimental set-up has been constructed. It consists of a cylindrical mould made from low-alloy steel which is insulated at the bottom and the top so that solidification occurs axisymmetrically. The radial temperature distribution in the mould as well as in the cast liquid is measured during the solidification process. Simultaneously the movements of the mould walls and the outer surface of the casting are measured. More information about the experimental method is available in earlier work.<sup>[58,59]</sup>

From the recorded temperature distribution a heat-transfer coefficient could be deduced as a function of time. The heat-transfer coefficient has a high value before an air gap has started to form. This value is determined by the contact resistance from mould roughness, coating, possible oxide layer of the melt etc. When an air gap starts to form, the heat-transfer coefficient decreases and is controlled by the conduction of the gap in the case of aluminium-base alloys.

In order to find a criterion for crack formation one has to define a so-called coherence temperature, a temperature where the metals starts to behave as a solid. In most models this temperature is set equal to the temperature where the metal is totally solid but this has been shown not to be correct. Data concerning the thermo-mechanical behavior of materials at high temperature, and especially during solidification are missing. At temperatures close to the melting point of metals, the stress-strain curve shows a strong dependence on the strain rate. Another important feature is the brittle behavior in a temperature range close to and somewhat below the melting point. Most theories presented in the literature are based on hot cracks being formed when liquid is present in interdendritic areas and at grain boundaries, under tensile stress. The temperature range when hot cracking occurs is therefore, related to the solidification interval and will be affected by segregation of elements. In contrast, many investigations have shown that hot cracking can occur below the solidus temperatures. It has also been shown that the cooling rate changes the ductile/brittle transition temperature depending on the solidification mode.

In the network there was discussion of the experimental methods to investigate a material's susceptibility to hot-crack formation. Two different methods have been used for comparison. One used conventional Gleeble testing equipment. In this method, a series of Al-base alloys were investigated. The work was performed by Alcan and the University of Swansea.<sup>[60,61]</sup> In the other method, a mirror furnace with ellipsoidal mirrors was inserted in a testing machine. The mirror furnace gives a limited heating zone at its focus. This makes it possible to melt a small part of the sample and let it solidify and cool to the test temperature. It was clearly shown that the material was brittle to a temperature far below the solidus temperature.<sup>[62,63]</sup>

Four programs have been available to the partners for dealing with air gap and hot crack formation. At the Foundry Institute at RWTH in Aachen, MAGMA and CASTS-SPAN3D are used. MAGMA is a commercial code whilst CASTS-SPAN3D is an in-house code developed at the institute.<sup>[64,65]</sup> The Technical Research Centre of Finland, VTT in Espoo, uses PROCAST which is a commercial simulation program for casting. The Centre for Material Processing CEMEF, Ecole des Mines de Paris, uses THERCAST which is a commercial code developed by CEMEF in co-operation with Transvalor.<sup>[66]</sup> The calculations have been performed with a large number of different boundary conditions. For a constant heat-transfer coefficient the programs all show the same results. The models for calculation of thermal stresses are different between the programs and thus the results are also. This work will be presented in a joint publication which is expected early in 2003.

As evident in the above description, this network benefited from some interdisciplinarity and from the fact that several materials were considered. This proved to be quite effective in improving exchanges of information. Common themes

from the network that appear to be of sufficient interest to warrant further projects are thermodynamic data and thermophysical properties, process control of microstructure, and virtual material design. It is worth stressing that basic research is needed also, i.e., that significant progress in understanding and modeling cannot be expected from applied research only. Both academic and industrial benchmarks should be developed to force improvement of the numerous simulation softwares developed nowadays. One typical challenge should be the description of the mechanical properties of partly solidified materials: designing experimental tests as well as challenging the emerging multi-phase codes. Further information on the MEBSP network can be obtained from the coordinator, at jacques.lacaze@ensiacet.fr, and for specific topic from the relevant coordinators. The final report will be available from the European Commission.

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