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Materials Characterization 46 (2001) 155–161

MATERIALS CHARACTERIZATION

Characterization of semisolid materials structure

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Received 27 December 2000; accepted 22 January 2001

Abstract

A special tool, which offers direct visualization and measurement of complex structures and 3D relationships, was developed. Thixomet image analyzer capabilities for 2D and 3D materials characterization with examples of commercial aluminum-based semisolid materials (SSM) are demonstrated. The study revealed that the structure of these materials had been totally skeletonized. Therefore, a multiply connected skeleton of the structure is a key attribute of semisolid-forming (SSF) technology. Such a skeleton is an inherent characteristic for all SSM suppliers. Many technological problems cannot be solved without an understanding of the nature of skeleton formation and its evolution at all stages of SSM production. Thixomet software performs a total quantitative description of 2D and 3D materials structures, including 3D parameters of skeleton and real morphology of alpha grains. © 2001 Elsevier Science Inc. All rights reserved.

Keywords: Semisolid materials; Structure; Characterizaion; Skeleton

1. Introduction

Advantages of semisolid-forming (SSF) technology in comparison with conventional casting methods, as well as its considerable progress, are well known [1], but there are still many problems hindering the full application of this technology. In order to solve these problems successfully, it is necessary to develop a special tool for objective quality estimation of semisolid materials (SSM) structure at all stages of their production.

Al–Si alloys, such as A356/357, are widely applied in SSF technology. The structure of these alloys consists of alpha solid solution aluminum grains surrounded by the alpha-Si eutectic. The SSF technology is based on the peculiarities of the rheological behavior of SSM. In turn, the rheological characteristics depend on alpha solid solution morphology. Therefore, the characterization and control of alpha solid solution morphology at each stage of SSF technology are of prime importance for a successful realization of this technology.

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2. SSM structure

The SSF technology will be discussed in respect to the requirements that can be imposed on image analysis in quality estimations of SSM structures.

2.1. Rheocasting

Rheocasting is the first stage of SSF technology when a thixotropic (nondendritic) structure is obtained through magnetohydrodynamic stirring of the alloy at the solidification front. It is only rheo-

1044-5803/01/$ – see front matter
PII: S1044-5803(01)00118-8

casting that lays the ground for the further evolution of the structure at other stages of SSF technology. There must not be typical dendrites in the structure of the billet. The fine thixotropic grains have to be minimally agglomerated, and the silicon distribution across the billet diameter has to be uniform.

2.2. Reheating

Reheating is the next stage of SSF technology when the slugs are heated in the induction furnace. During the reheating cycle, the important globularization of the thixotropic alpha solid solution grains occurs. Associated negative processes, such as structural coarsening, formation of occluded eutectic, and further structural skeletonization, which started during rheocasting, also take place here. The chosen reheating temperature–time parameters must promote globularization of the thixotropic grains while limiting, as much as possible, the detrimental processes.

2.3. Forming

Forming is the final stage of SSF technology, which is entirely dependent on the previous stages. In the casting machine, the ram injects the slug into the die cavity and applies the pressure to produce the thixotropic castings.

The above-enumerated special features of SSF technology produce the microstructural features, which establish the requirements placed upon the Thixomet image analyzer for the estimation of the quality of the SSM structure.

3. The principal capabilities of Thixomet for the characterization of SSM structure

Thixomet provides the special operations for characterization of SSM structure as well as the standard operations applied in commonly used image analyzers. The following special operations are presented for discussion.

3.1. Evaluation of the distribution of porosity, eutectic content (continuous and occluded), and silicon content across the specimen

The silicon content is calculated from the eutectic content by the rule of the segments (Eq. (1)):

$$\mathrm{Si\ wt.\%} = \mathrm{b} + \mathrm{Q_{eu}}(\mathrm{d} - \mathrm{b}), \tag{1}$$

where b is the silicon solubility in the alpha solid solution at the eutectic temperature, d is the silicon content at the eutectic point of the phase diagram, and Q_{eu} is eutectic fraction determined by image analysis.

3.2. Evaluation of special shape factor of the grains

A special grain shape factor is calculated in studies of anodized specimens in polarized light. The identification and integration of the dendrite's fragments, revealed in the planar image, into one structurally isolated object is possible as a result of this measurement. A dimensionless grains' shape factor F_{g} represents the area of a specific interface surface relative to that of one structurally isolated grain [2]:

$$F_{\mathrm{g}} = \frac{1}{6\pi}\frac{S_V^2}{f_z N_A}, \tag{2}$$

where N_A is the number of structurally isolated grains per unit area, f_z is the volume fraction of alpha solid solution grains, and S_V is the area of the alpha solid solution–eutectic interface per unit volume. Referring to Ref. [3], the magnitude of S_V is proportional to the length of boundary lines of alpha solid solution L_{zl} on the microsection with the area A (Eq. (3)):

$$S_V = \frac{4}{\pi}\frac{L_{zl}}{A}. \tag{3}$$

Therefore, the shape factor of the thixotropic grains can be evaluated taking into account the structural connections between its fragments. In this approach, the total specific length of interface boundaries is a function of the number of identified structurally isolated objects and not to the overall number of grains revealed in a given field of view. The grains are grouped on the principle of their identical crystallographic orientation. This fact is fixed by the common color of these grains. Formal parameters by which the objects are combined with each other are the grayscale level of these objects and the distance between them (Fig. 1). As follows from these results, the overall number of the grains revealed in the given field of view is 427, but the number of identified structurally isolated objects is 147 only. The corresponding values of shape factor are $F_{\mathrm{g0}} = 17$ and $F_{\mathrm{g}} = 43$. The identified fragments of dendrite of structurally isolated object and corresponding parameters can be found in lower right-hand corner of Thixomet's window.

3.3. 3D reconstruction of the structure

3D structural reconstruction by sequential polishing of the SSM has been carried out to check the

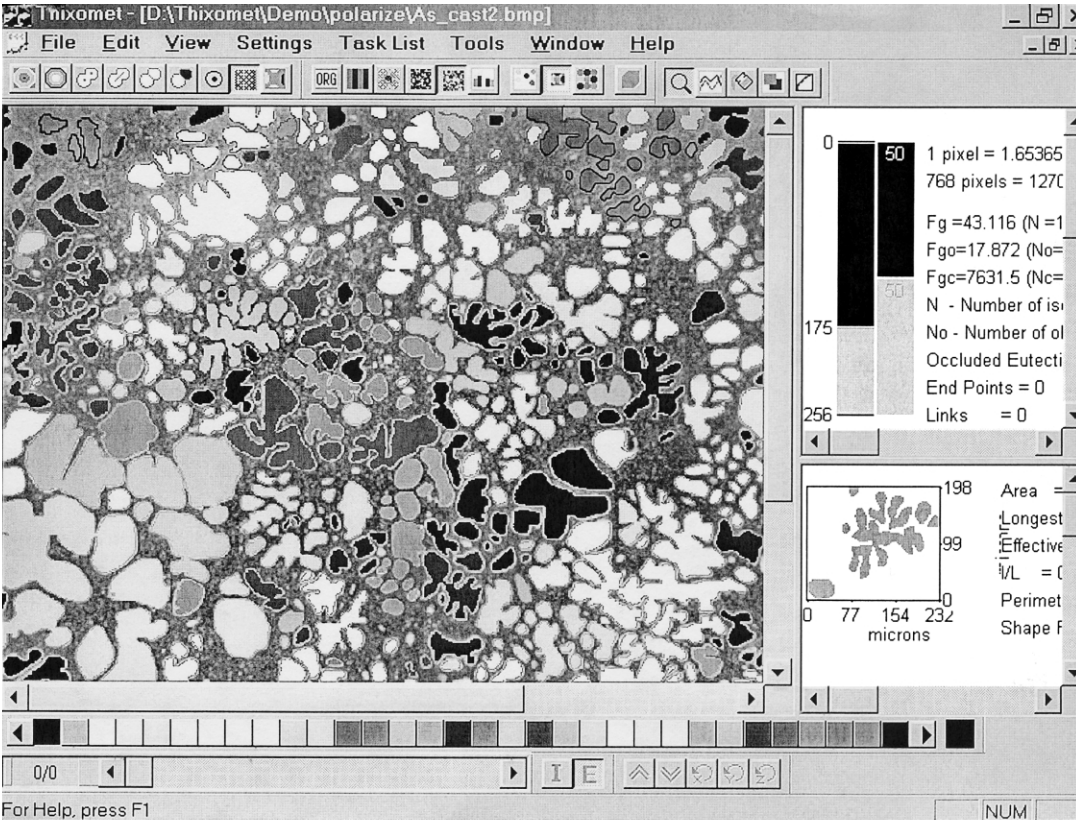

Fig. 1. Thixomet screen. Identification and integration of dendrite's fragments into structurally isolated object.

adequacy of the grain morphology estimation using the shape factor F_g from the Eq. (2). The images of the structure (with previously placed microhardness indentations for registration) were saved as graphic files. These files were used for precise positioning of the planar images in 3D space. The spacing between adjacent images was calculated by the change in the indentation size with polishing cycles.

To reconstruct the real picture of the 3D space connections of the grains, the processing of binary images was carried out as follows:

- A Euclidean Distance Map (EDM) was constructed of both the grains and the background (Fig. 2). From the EDM, topological characteristics of the objects (end points, links, loops, and branches) and a skeleton were determined to obtain the morphology

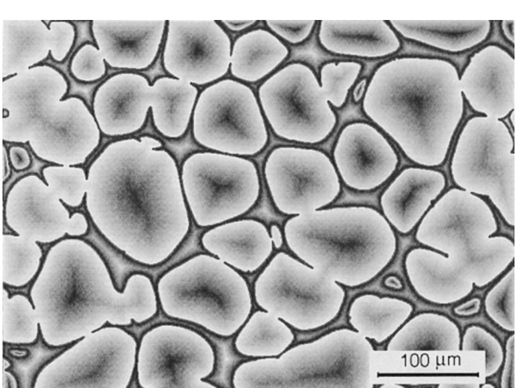

Fig. 2. EDM of structure.

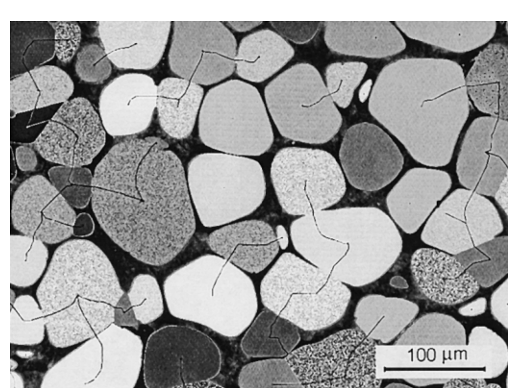

Fig. 3. Separating of touching grains and 2D skeleton.

information. The EDM of the background containing the information on object clustering could be used to determine the criteria of uniform distribution of the objects across the microsection [4].

- Separating of touching grains and 2D skeleton of the objects were carried out (Fig. 3).

The results of these studies were sufficient for the development of a unique 2D presentation procedure of the 3D reconstruction results.

3.4. Evolution

Evolution is the procedure that works with the stored images of sequentially examined sections. These images are loaded into the PC memory. Using the scroll bar, the user outputs the sequential images to the Thixomet main window. As the adjacent images are replaced, the grains' configurations and their colors are changed. The user observes these changes according to the true connection of the grains in 3D space: the grains that are connected with each other in 3D space are the same in color and the user watches the color expansion. Thus, as the number of analyzed images changes, the structure connectivity extends and corresponding shape factor value of the grains also changes.

3.5. Identification

Identification is a procedure that also works with the stored images of sequentially examined metallographic sections. However, the user observes only the first image of the first field of view, that is, the grain configuration is fixed during the analysis. Using scroll bar, the stored loaded images of the sequentially examined sections are analyzed step-by-step within the PC memory. As a result of this analysis, the user observes the expansion of colors in accordance with the real connectivity of the grains in 3D space. Thus, as the number of analyzed sections changes, the connectivity of the structure extends, and corresponding shape factor value of the grains also changes.

The analysis of the real connectivity of the grains in 3D space by the evolution and identification procedures revealed that the method of grains' integration, according to the criterion of identical crystallographic orientation, is not sufficient. This is explained by the dual nature of thixotropic grains' connectivity, namely, structural and technological. The structural nature relates to the identical crystallographic orientation of the grains, inherited from the dendrites. The technological nature of the grains connections relates to the collisions and welding, Ostwald ripening, and coalescence of separate thixotropic grains into a common skeleton at all stages of SSF technology. The technological nature of the grains' connections cannot be identified only by the crystallographic orientation criterion.

Therefore, the studies of anodized specimens in polarized light can be carried out only for the estimation of the structural connection of the grains and for the as-cast state only. These studies cannot be applied to the investigation of the samples in the as-reheated and as-formed conditions. The connection of the grains in these cases relates mainly to their technological nature. Therefore, the grains' shape factors and the extent of their connectivity can be adequately estimated only from the results of the 3D reconstruction of the structure.

3.6. 3D skeleton

The next step towards image processing in 3D is the determination of the 3D skeleton that simplifies the structure. The skeleton in 3D is formed by connecting the ultimate eroded points of the 2D sections (Fig. 4). It correctly depicts the topological properties of the structure closely related to the shape parameters. The connectivity of a skeletal structure is a topological property that is directly related to rheological properties. Since our first studies of the 3D skeleton were carried out, a multiple connected skeleton was found in the structure of SSM. Multiple connectivity denotes that some nodes may be connected to another by more that one path through the skeleton.

For the quantitative estimation of connectivity of multiply connected structures, the first Betti number of the network (in our case the network is the skeleton), p_1, is best to use [5]. This number is defined as the number of branches in excess of the number required to form a tree ($p_1 = 0$) between the nodes in the skeleton [5] (Eq. (4)):

$$p_1 = b - n + p_0, \tag{4}$$

where b is the number of branches, n is the number of nodes, and p_0 is the number of disconnected skeletons (first Betti number). Thus, the connectivity of the

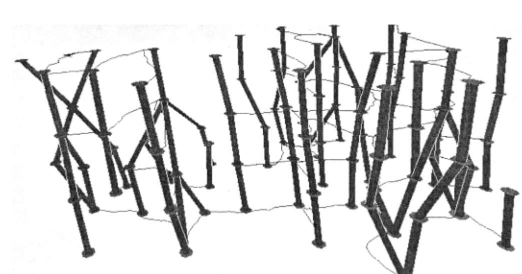

Fig. 4. 3D skeleton zoomed by z-direction.

structure is the number of branches that could be removed from the skeleton without creating additional separate parts.

3.7. 3D reconstruction of the grains surface

Ito et al. [6] have already described the single agglomerate of alpha grains present in thixotropic Al–6.5 wt.% Si alloy specimens, which were obtained by mechanical stirring under laboratory conditions. To reconstruct the 3D structure of the agglomerate, a set of photographs was used in this study. In another work [7], the detailed computerized 3D reconstruction of the structure by microstructural tomography and analysis of the skeletal network was presented. The authors investigated the Ostwald ripening mechanism of Sn particles in a Pb–Sn eutectic. But, such 3D reconstructions for the commercial SSM have not been carried out until now.

The final stage of image processing in 3D is 3D reconstruction of the grain surfaces with the possibility of the analysis and presentation of the real surfaces of the grains (Fig. 5).

The capabilities of the Thixomet 3D software can be summarized as follows:

- 3D structural reconstructions using sequential planar examination;
- all operations with 3D image viewing (rotation, shifting, enlargement);
- 3D measurements of the objects;
- computation and visualization of any profile of the 3D objects at the intersection with any sectioning plane;
- image import–export into standard 3D format.

4. Investigation of SSM

Thixomet capabilities have been used for comparison investigations of commercial SSM.

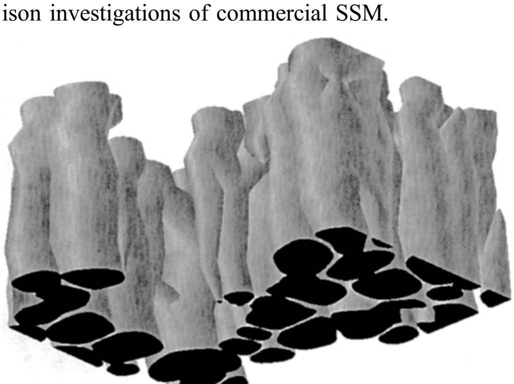

Fig. 5. Reconstruction results of real surfaces of grains (zoomed by z-direction).

4.1. Materials and method

The samples were obtained from three companies (suppliers of SSM). Let us represent them as Suppliers 1, 2, and 3 and their samples were coded as 1, 2, and 3, respectively. The 357 alloy samples for the studies measured $15 \times 15 \times 76$ mm and were machined from the 76-mm diameter billet. All studies were carried out in the as-cast state at $\times 200$ magnification except for two samples from Supplier 1 (sample codes 1-2 and 1-A), which were in the as-reheated state, and were examined at $\times 100$. The reheating procedure was as follows: the samples were held for 8 min at 587°C and quenched in water.

Metallographic imaging used the reflected light provided by an MeF3A Reichert–Jung metallographic microscope with field positioning based upon microhardness indents. Up to 50 sequential parallel planes-of-polish were examined on each specimen using conventional specimen preparation with a Buehler Automet device. The thickness of the material removed was 29–42 μm for the as-cast specimens and 70–123 μm for the as-reheated specimens.

4.2. Results and discussion

The investigations of the nature of the silicon distribution across the billet's section revealed the distinctive segregation in Specimen 3 in comparison with Specimens 1 and 2. The increased silicon content inside the near-surface layer of the billet can cause the eutectic to flow out of the slug during reheating. Besides, with increasing of silicon content in local zones, the temperature, at which the melting of alpha solid solution begins, rises, and the process of eutectic melting during reheating slows down. As a result, there are coarse silicon crystals in such zones of the casting.

The determination of the connectivity of grains based upon a common color, was carried out by the procedures of evolution and identification. These results are sufficient to allow definite conclusions as to the structural quality of the investigated samples (Fig. 6). The grains are artificially textured in accordance with their real 3D connections. The same texture of the grains denotes that they are integrated into one skeleton. The marginal and isolated single grains are colored white.There is a minimum structural connectivity ($p_1 = 27$ or 2.2×10^4 l/mm^3) in Specimen 3. Thirteen fine skeletons (10.6×10^3 l/mm^3) were revealed inside this sample and all grains were rather uniformly integrated into six separate bigger skeletons (Fig. 6a). Skeleton connectivity was proportional to skeleton size: the greater the number of

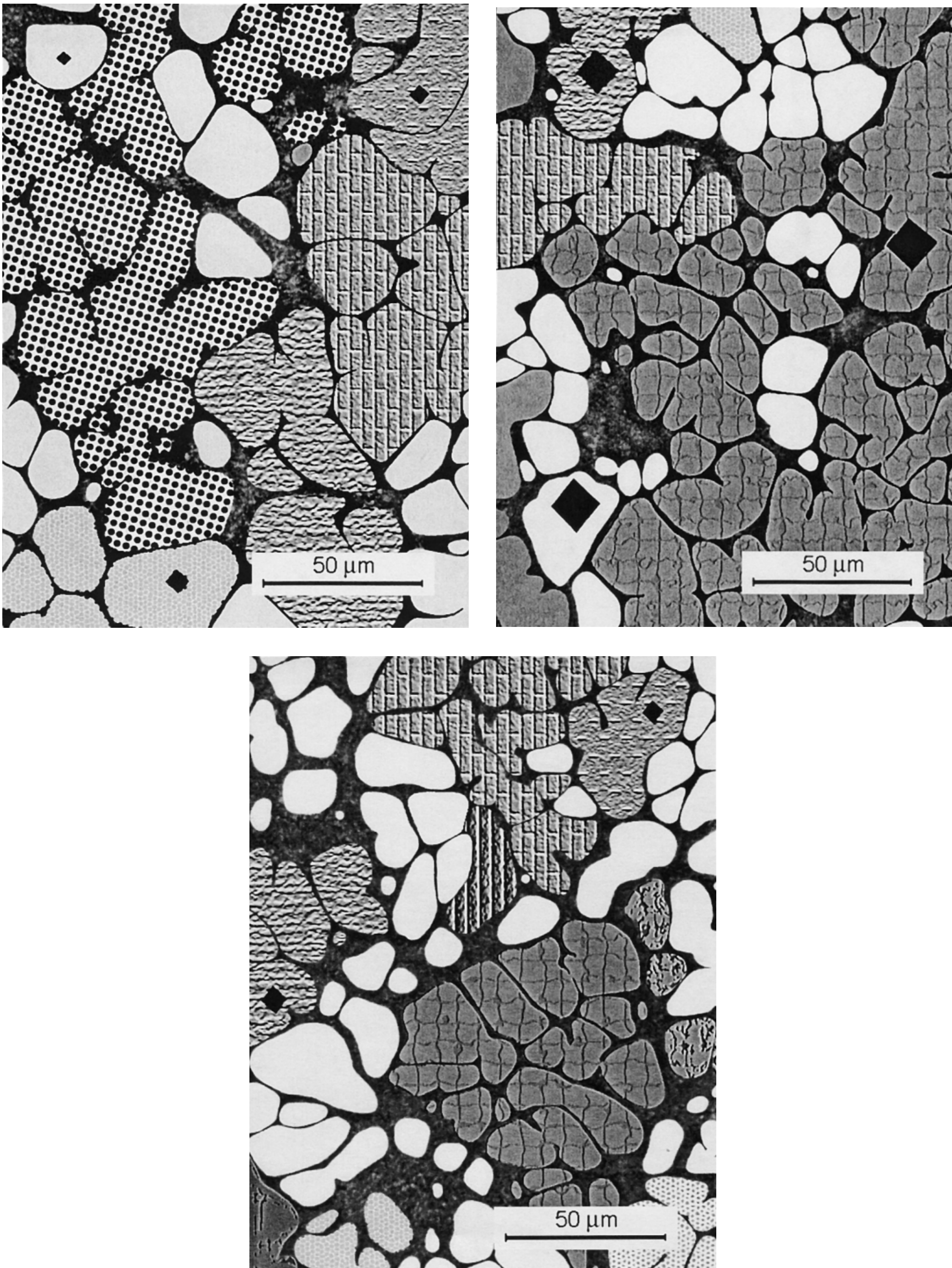

Fig. 6. Determination of grains connectivity by the evolution procedure, last images. (a) As-cast Specimen 1, depth of analysis is 41 μm. (b) As-cast Specimen 2, depth of analysis is 28 μm. (c) As-cast Specimen 3, depth of analysis is 42 μm.

grains integrated into the skeleton, the higher the multiple connectivity.

Specimen 2 was the most multiply connected ($p_1 = 48$ or 5.8×10^4 l/mm^3) as most of the grains (68%) were integrated into one multiply connected ($p_1 = 45$) skeleton (Fig. 6b).

Specimen 1 was intermediate in structural quality between Specimens 2 and 3. A large part of the excess connections (22 of 29) in this sample, along with Specimen 2, was accounted for by one huge skeleton, which combined 47% of the total number of grains (Fig. 6c). The rest of the grains were sufficiently uniformly distributed among five small skeletons that had few excess connections. Thus, if a structure with minimum skeletonization and minimum multiple connectivity is necessary to ensure rheological properties for SSM, the as-cast samples would be arranged in following decreasing order — 3, 1, 2 — as to their suitability for SSF technology.

There are evident changes in the structure of the specimens after reheating. With regards to microscope magnification and polishing depth, the investigated volume of the specimens in as-reheated state was 12–28 times larger than the volume of the specimens in as-cast state. Specific connectivity of the structure in as-reheated state was many factors of 10 smaller than in as-cast state because of structural coarsening and Ostwald ripening processes. It should be noted that eutectic quantity has a determining influence upon the skeletonization process and the formation of excess connections during reheating. The decrease in the eutectic content from 31% to 37% in Specimen 1-A to 21–23% in the Specimen 1-2 causes the structure of Specimen 1-2 to be entirely skeletonized: 88–96% of all grains are combined within a single multiply connected skeleton: $p_1 = 34–37$ or 0.25×10^4 to 0.27×10^4 l/mm^3.

5. Conclusion

The study revealed that SSM structure has been totally skeletonized. Therefore, a multiply connected skeleton of the structure is a key attribute of SSF technology, which greatly influences the rheological properties of SSM. Such a skeleton is an inherent characteristic for all SSM suppliers. Many of SSF technology problems cannot be solved without an understanding of the nature of skeleton structural formation and its evolution at all stages of SSM production.

Thixomet software performs a total quantitative description of 2D and 3D SSM structures, including 3D parameters of structure skeleton and real morphology of alpha grains. We developed a special tool, which offers direct visualization and measurement of complex structures and 3D relationships.

The next step in growth of Thixomet approach will be to further develop criteria and standards for quality estimation of SSM 3D structures for industrial application. Such an approach will permit us to carry out analysis of SSM quality at all production stages. The results of this analysis will be useful for SSF technology improvement.

Acknowledgments

The authors gratefully acknowledge the contribution to this research of Daniel Adenis, who was also an initiator of work in SSM structure characterization. Special thanks are due to Svetlana Pavlova and Elena Kazakova, who helped carry out metallographic investigations.

References

[1] Midson SP, Brissing K. Semi-solid casting of aluminum alloys: a status report. Mod Cast 1997;2:41–3.
[2] Loue WR, Suery M. Microstructural evolution during partial remelting of Al–Si7Mg alloys. Mater Sci Eng, A 1995;203:1–13.
[3] Saltykov SA. Stereometric metallography. Moscow: Metallurgizdat, 1958. pp. 167–75.
[4] Russ JC. The image processing handbook. 2nd ed. Boca Raton (FL): CRC Press, 1995. pp. 476–80.
[5] De Hoff RT, Aigeltinger EH, Craig KR. Experimental determination of the topological properties of three-dimensional microstructure. J Microsc 1972;95: 69–91.
[6] Ito Y, Flemings MC, Cornie JA. Rheological behavior and microstructure of Al–6.5%Si alloy. Proceedings of the Symposium on Nature and Properties of Semi-Solid Materials, San Diego, CA, March 1–5. Minerals, Metals & Materials Society, 1991. pp. 3–17.
[7] Wolfsdorf TL, Bender WH, Voorhees PW. The morphology of high volume fraction solid–liquid mixtures: an application of microstructural tomography. Acta Mater 1997;45(6):2279–95.