Our Understanding of Macrosegregation: Past and Present

Merton C. FLEMINGS

Toyota Professor of Materials Processing, Massachusetts Institute of Technology, Cambridge, Massachusetts, USA.

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Macrosegregation has long been a topic of engineering importance, with recorded publications on the subject extending back nearly half a millennium. The phenomenon has assumed new importance in recent decades with the advent of new casting processes including continuous casting. This lecture reviews our current understanding of macrosegregation, with particular reference of carbon steel ingots and continuous castings. Aspects of the problem are noted where our understanding remains weak.

KEY WORDS: macrosegregation; solidification; ingots; continuous casting.

The Historical Understanding

That macrosegregation occurs in castings and ingots has been understood for centuries. Biringuccio, as early as 1540, described segregation in bronze gun barrels.¹⁾ Much literature on the subject may be found, published over the succeeding 460 years. By the turn of the current century, as steel came into widespread use as a structural material, failures began to be observed, attributable to macrosegregation, and researchers began to focus on the problem, its causes and its solution.

The cause of positive macrosegregation (increase in alloy content above nominal) in the central portions of steel ingots seemed obvious. As Bradley Stoughton wrote in 1911 in his widely read book “Metallurgy of Iron and Steel”:²⁾

“...It can readily be conceived how each layer that freezes, beginning at the outside, rejects some of its impurities to be dissolved by the still liquid mass in the interior...so that as a general thing the portion of the metal richest in impurities...will be that which freezes last...evidently just below the bottom of the pipe”.

What could be more reasonable, even self-evident? The advancing solid front is purer than the liquid and so it pushes the solute in advance of the front to the last place to freeze, the bottom of the pipe. The view was so obviously sensible that it was to remain the teaching of the field for another fifty years. For example, John Bray in 1942, in his book “Ferrous Production Metallurgy”³⁾ wrote a nearly identical paragraph on the subject:

“It is easy to see how the process of solidification would result in isolation of certain components...forming in the ingot (usually at the bottom of the pipe) a solid mass that is very different in composition”.

The basic view of these authors persisted widely in the literature up to the 1960’s, although other ideas were some-

times expressed with the conviction of fact. For example, the third edition of “Basic Open Hearth Steelmaking,”⁴⁾ 1964, implied that the only forms of macrosegregation were “A” and “V” segregates, **Fig. 1**, and that the former came from the settling of crystallites which forced impure liquid upwards to be periodically entrapped by the growing solid front. Until the latter part of the 1960’s, no serious attempt was made to prove the correctness of the various explanations of macrosegregation in steel, or to make them quantitative.

Of course, commercial interests, particularly the desire to maximize “yield” of salable product from each ingot, led to extensive empirical studies on ingot design to minimize

Fig. 1. Macrosegregation in a killed carbon steel ingot. Segregation is positive in the upper and central regions, negative at the base. Concentrated “A” and “V” segregates are shown. Lighter “bands” near the periphery are not shown.⁴⁾

Fig. 2. A "volume element" from the liquid-solid zone in a solidifying ingot. (a) The simple microsegregation model, (b) incorporating interdendritic fluid flow.

segregation and pipe. A good example is the work of Gathman⁵⁾ who obtained 85 patents on ingot design between 1909 and 1942 and whose designs were employed by most major U.S. steel producers. Important empirical research also focused on detailed examination of ingot structures. Notable examples are the 1937 report of the Iron and Steel Institute, London, on the heterogeneity of steel ingots⁷⁾ and Marburg's article of 1953.⁶⁾ The "textbook" schematic diagram of steel ingot macrosegregation that emerged is illustrated in Fig. 1, showing a broad region of centerline segregation, concentrated at the bottom of the "pipe", "A" segregates, and "V" segregates. Banded segregates, particularly along the outer periphery of the ingot, were also known to exist, but were less of a problem since the compositional variations in these bands were not large. A negative "cone" of crystallization was understood to exist toward the bottom of the ingot. This was widely assumed to come from falling crystallites, although serious questions were raised regarding the feasibility of this mechanism.^{6,7)}

In this talk, I will summarize the steps leading to the quantitative understanding of macrosegregation in castings and ingots we have today, discuss the application of these ideas to carbon steels, and then comment on what we do not yet understand well about macrosegregation. The understanding is, of course, very different from that of the older textbooks. In a sentence, our current understanding of macrosegregation in all metal alloys can be summarized as follows:

"All types of macrosegregation in castings and ingots form within the liquid-solid zone. In most cases, it is the result of slow interdendritic flow, driven by shrinkage, geometry, solid deformation or gravity. In some cases it may be the result of solid movement in the early stages of solidification. (*i.e.*, settling)".

Microsegregation

An essential foundation of the development of a quantitative macrosegregation theory was the formulation of a sound microsegregation theory. Microsegregation was, in

fact, well treated many years ago by Scheil⁸⁾ employing a model essentially that of Gulliver⁹⁾ from as early as the 1920's. The model assumes complete diffusion in the liquid over the size scale of the dendrite arm spacing, no solid diffusion, no undercooling before nucleation or from curvature effects, no ripening during growth, no macroscopic mass transport, no pore formation. It is written in various forms describing either the evolution of the solid composition that forms during solidification, or evolution of the interdendritic liquid composition.¹⁰⁾ It is written here for a binary alloy with partition ratio k , as the change in interdendritic liquid composition, C_L , with change in local mass fraction liquid, f_L :

$$\frac{df_L}{dC_L} = - \left(\frac{1}{1-k} \right) \frac{f_L}{C_L} \dots\dots\dots (1)$$

The "local mass fraction liquid" is that within a "volume element" such as that sketched in Fig. 2(a), comprising some small number of dendrite arms. The simple relation of Eq. (1) can be readily modified to account for diffusion in the solid, coarsening, undercooling before nucleation, and the macroscopic liquid diffusion in the growth direction. It can also be extended to apply to multi-component alloys. These equations are termed the "local solute redistribution" equations or more simply "solute conservation" equations. One or another of these solute conservation equations forms the starting point of all modern macrosegregation analyses.

A period of some confusion concerning the validity, and certainly the range of validity of the Scheil relation occurred in the 1950's and early 1960's following studies showing the solute buildup in front of the growing solid-liquid interface in single crystal growth and the resulting potential for "constitutional supercooling."¹¹⁾ It was only in the latter part of the 1960's that it became clear that dendritic growth reduces such undercooling to a negligibly small value, making the relation of Eq. (1) (and its modifications referred to above) valid descriptions of microsegregation in usual casting and ingots.

Fig. 3. Schematic diagram of apparatus employed to measure flow rate through the semi-solid alloy. (From Ref. 13.)

Interdendritic Fluid Flow

A second essential foundation for a macrosegregation theory is a quantitative understanding of interdendritic fluid flow behavior. It has long been known that feeding of solidification shrinkage in dendritically freezing alloys takes place first by “mass feeding” when equiaxed dendrites are still free to move. Then, in later stages of solidification, it was understood that fluid flow must take place through a stationary solid dendritic network.¹²⁾ Based on the solidification model described above, it was reasonable by the 1960’s to assume that the interdendritic fluid flow would follow the same relations observed for other types of porous media. This was first shown experimentally in 1966, in an experiment in which molten lead was forced through the dendritic semi-solid network of an aluminum–copper alloy, and flow rate measured for various pressures and fractions solid.¹³⁾ At higher fractions solid, data were also obtained by passing nitrogen directly into the semi-solid sample, forcing out the interdendritic liquid. The apparatus employed in that initial experiment is shown in Fig. 3. Experimental results, over a wide range of fractions solid, were found to obey the classical Darcy’s Law that may be written for the horizontal, unidirectional flow involved as:

$$v_x = -\frac{K}{\mu g_L} \frac{dP}{dx} \dots\dots\dots (2)$$

where K is “permeability”, dP/dx is pressure gradient, μ is viscosity, and g_L is volume fraction liquid (nearly, but not quite, mass fraction). Figure 4 shows results of that early experiment.

Since interdendritic fluid flow influences macrosegregation directly, there is much interest in experimental measurements today of permeability, and on mathematical models of how permeability varies with fraction solid, coarsening, columnar vs. equiaxed structures, etc. No attempt was made in the original study above to control dendrite arm spacing or to compare measured permeabilities

Fig. 4. Permeability versus fraction solid for Al-4.5%Cu alloy. Circles represent data obtained using nitrogen gas, and crosses represent data using molten lead.

with calculation. Much subsequent work, however, has done so. For example, Poirier and Ocansey¹⁴⁾ measured permeability in equiaxed dendritic structures and compared a measured dimensionless permeability (permeability times specific surface area squared) with that calculated. They showed that their results and those of Murakami and Okamoto¹⁵⁾ compared favorably with an analysis of Zick and Homsy¹⁶⁾ developed for Stokes flow through an array of spheres. Permeability is usually assumed to be independent of direction and this appears to be a reasonable approximation even for columnar structures at the higher fractions liquid.¹⁷⁾ At the lower fractions liquid, however, permeability is expected to depend on orientation, given the plate-like nature of dendrite formation as solidification progresses. Numerical values of permeability employed in macrosegregation calculations today are usually obtained from experiment, calculation or a combination of both and may or may not take into account the ripening that takes place during solidification.

The Impossibility of the Old Steel Macrosegregation Model

By 1967, a good working model was in hand for dendritic growth in steel and other metals, as diagrammed in Fig. 5. Solidification was understood to be either columnar or equiaxed, with interdendritic liquid composition and fraction liquid at any point determined by Eq. (1) (or similar formulation as discussed above) and the relevant alloy equilibrium phase diagram. Whether equiaxed or columnar, after a certain stage of solidification the liquid–solid zone was understood to comprise the closely spaced dendritic network sketched in Fig. 2(a).

For the solidification rates and gradients involved in usual ingot and continuous casting processes, in 1967 we understood that only negligible solute buildup could occur at the dendrite tips in steel castings and ingots. Hence, significant solute from this source could not be “pushed” to the ingot center, or carried there by convection. It was also clear that the classical explanations could not account for “A” segregates, or for “V” segregates. Even the best of the

Fig. 5. Model of the liquid–solid region showing distribution across the liquid zone of a) dendritic structure, b) temperature, c) liquid composition, d) fraction solid.

explanations extant no longer seemed probable, an example being that for “V” segregates in the Seventh Report on Heterogeneity in Steel Ingots of 1937.⁷⁾

“The centre “V” segregate is presumably caused by the impurities in the enriched mother-liquor arranging themselves more or less in horizontal zones (perhaps by gravity), these zones being subsequently distorted into the characteristic “V” form by the contraction of the last metal to freeze.”

Finally, in the nonferrous literature, there were, by 1967 excellent studies showing that at least two types of segregation could result from interdendritic fluid flow: “exudation” and “inverse segregation.”^{13,18)} And so the stage was set for the modern macrosegregation theory.

The Beginning of Modern Steel Macrosegregation Theory

The experiment which demonstrated, in 1967, how interdendritic flow could cause macrosegregation other than inverse segregation or exudation was essentially that illustrated in Fig. 6.¹⁹⁾ An aluminum alloy casting of vertical cross section shown was unidirectionally solidified from the base upwards. Figure 6(a) shows schematically the liquid–solid region at the moment the growing dendrites reached the reduced cross section. The solidus is a distance ($x_r - x_e$) back from the reduced cross section.

Up until the moment shown in Fig. 6(a), interdendritic fluid flow to feed shrinkage will be all unidirectionally downwards. However, once the tips reach and go above x_r , flow to the outer reaches of the broad cross section must have a horizontal component, and necessarily less of a vertical component. Meanwhile, vertical downward flow just above x_r must be much greater than that just below, since flow to feed the large cross section must now come from a much smaller cross section. Streamlines for the flow after the tips protrude into the reduced section are shown

Fig. 6. Macrosegregation in a unidirectionally solidified casting of reduced cross section. Schematic diagrams of (a) solidification when location of the reduced cross section is just below the liquidus temperature, (b) flow lines for feeding solidification shrinkage at this time of solidification, (c) resulting macrosegregation.

schematically in Fig. 6(b).

The components of flow in the y and z directions would be expected to have no effect on macrosegregation, since there is no variation of liquid composition in these directions. Flow in the x direction, however, must have an effect, since liquid composition does vary in this direction. On examination of the solidified casting, marked positive segregation was found over most of the region ($x_r - x_e$), excepting only that central portion just under the reduced cross section. Marked negative segregation was found in the reduced cross section just above x_r . This segregation is shown schematically in Fig. 6(c). Thus, this experiment, and a number of others which followed, pointed clearly to interdendritic flow perpendicular to isotherms as the central fac-

tor in determining macrosegregation in steel castings and ingots.

The Rudimentary Theory

Quantitative treatment of macrosegregation begins with treating the volume element of Fig. 2 as a differential element and examining the effect of flow on the local solute redistribution. The result is an equation modified from that of Eq. (1) by substitution of volume fraction liquid, g_L , for mass fraction liquid, f_L , incorporation of solidification shrinkage, β , and of (v_x/R) , the component of flow in the x direction, divided by the velocity of the planar isotherm perpendicular to, and moving in, the x direction. The resulting solute conservation equation for this case is written:^{20,21)}

$$-\frac{\partial g_L}{\partial C_L} = \left(\frac{1-\beta}{1-k} \right) \left(1 - \frac{v_x}{R} \right) \frac{g_L}{C_L} \dots\dots\dots (3)$$

Note that Eq. (3) reduces to Eq. (1) when v_x and β both equal zero; that is, when there is neither solidification shrinkage nor fluid flow. It also reduces to Eq. (1) when

$$v_x = - \left(\frac{\beta}{1-\beta} \right) R \dots\dots\dots (4)$$

This flow is just that required to feed solidification shrinkage in unidirectional solidification, and in steady state solidification the result is no macrosegregation. Flow greater (down the temperature gradient) with a speed than that of Eq. (4) results in negative segregation; a speed that is less, or in the opposite direction, results in positive macrosegregation.

Calculation of macrosegregation is done by recognizing that the solid composition forming during solidification is related to the liquid composition by the partition ratio, and then integrating Eq. (3) over the full range from the beginning to end of solidification to calculate the total final local solid solute content. The integration is simple when R is known or assumed. It is considerably more difficult in the more usually important case where R must be determined from a formulation of flow through porous media, and where the coupled problem must be considered.

Gravity Caused Convection

What became apparent soon after the formulation of Eq. (3) was that gravity, acting on a liquid of variable density, could be an important cause of macrosegregation. The density of the interdendritic liquid varies during solidification as a function of both temperature and solute content, and gravity acting on this liquid can cause quite significant flow differences from what would otherwise be expected. In fact, it was soon understood that gravity could even reverse the direction of expected flow of the X component. At this limiting case

$$\frac{v_x}{R} > 1 \dots\dots\dots (5)$$

and by inserting this relation in Eq. (3) it is readily seen that, although the temperature continues to fall (and so local liquid composition increases), local melting occurs, rather than solidification, *i.e.*, dg_L/dC_L becomes positive

and therefore dg_L/dT_L becomes negative. It is this local melting and the resulting flow instability that result in the channel segregates known in their various manifestations as “freckles” or “A” segregates, and no doubt contribute to “V” segregates. The basic relation of Eq. (5) was first understood qualitatively on the basis of laboratory experiments^{22,23)} and later interpreted analytically with the aid of Eq. (3).²⁴⁾

Figure 7 shows an early experiment on formation of these channel segregates. An aluminum–copper alloy casting was solidified unidirectionally, horizontally. In this alloy, interdendritic liquid density increases with increasing solute content and so the streamlines of the interdendritic flow are caused to bend downwards as shown in Fig. 7(c). At sufficiently slow solidification velocity, the flow lines not only were found to bend downwards, but looped to change direction, as shown in the lower right of Fig. 7(c). In this area the condition of inequality 5 was reached, and channel segregates were found.

A Qualitative Understanding of Macrosegregation in Castings and Ingots

The early work described above, reinforced by many subsequent studies, is sufficient to gain a qualitative understanding of the main forms of macrosegregation encountered in steel castings and ingots. Ingots are usually of rather tall and of sufficient cross section that solidification rate, after at least the first few inches of solidification, is rather low. Both factors accentuate the gravity driven convection and its effect on segregation. In the case of carbon steels, liquid density generally decreases during solidification resulting in a flow component vertically upward within the liquid–solid zone. The flow has an inward component because of the higher permeability of the liquid–solid zone in inner regions of the ingot, and often also because of ingot geometry. While a still fully liquid zone exists at the center of the ingot, interdendritic flow, except in the lower portions of the ingot, can therefore be expected to be primarily outward and upward. A simplified schematic of this is shown in Fig. 8(a). After the liquidus isotherms reach the center of the ingot, there must be also be a downward flow within the liquid–solid zone, Fig. 8(b). The result of the upward flow is positive segregation at the central and upper regions of the ingot. When the flow is sufficient to meet the condition of inequality 5, “A” segregates result along the flow path. Solute rich liquid can flow from the liquid–solid region into any remaining fully liquid region when “A” segregates are large enough that local equilibrium no longer exists between these and the surrounding dendrites.

When a hot top is too small or inadequately insulated its temperature can drop below that of the region just under the hot top. This, unfortunately has too often been the case in practice. In this situation, interdendritic liquid, flowing downwards to feed solidification shrinkage, passes through a positive temperature gradient, Fig. 8, and positive segregation necessarily occurs. Poor hot top design (by today’s standards) was most certainly a contributing factor to the macrosegregation observed by Bradley Stoughton and others “below the bottom of the pipe”.

The outward interdendritic flow at the bottom of the ingot, being down a temperature gradient must result in

Fig. 7. Unidirectionally and horizontally solidified ingot. Sketches of (a) ingot, (b) solidification, (c) interdendritic flow lines.

negative segregation in this region. It is quite possibly the primary cause of the well known "negative cone" of segregation. Crystallite settling may well contribute a portion of this negative cone of solidification, but at least in some cases the arguments against it are compelling, most notably when a negative cone of segregation is found in a portion of an ingot that is fully columnar.^{6,7)}

The light "bands" always seen near ingot surfaces upon careful etching are also readily explained. Some degree of macrosegregation always occurs in ingot solidification when isotherms are moving at anything other than "steady state". Thermal fluctuations, whether caused by variations of mold-metal interface resistance or by convective currents result in positive or negative "bands."²¹⁾

Segregation in continuous casting, at least at the steady state, is also simply summarized in the light of the model given here. If flow lines were perfectly straight in the cast-

ing direction, Fig. 9(a), no segregation would result. If flow lines deviated outwards, Fig. 9(b), negative segregation would result in inner portions of the casting. If flow lines deviated inwards, Fig. 9(c), positive segregation would result in the inner regions and "V" segregates could result if deviation were sufficiently great. Centerline segregation will be accentuated by casting "bulging" which will increase interdendritic fluid flow; any unsteady state perturbations of heat flow or casting speed will also alter interdendritic fluid flow and therefore macrosegregation.

Steel Macrosegregation Modelling

The early work outlined above was on binary systems, with simplified, uncoupled solution of the transport equations. More recently, models have been developed that are fully coupled numerical solutions of the mass, momentum, energy and solute conservation equations. Some of these

Fig. 8. Schematic diagram of flow lines which would lead to the main forms of macrosegregation observed in ingot solidification: (a) central fully liquid zone present, (b) later stage of solidification.

Fig. 9. Interdendritic fluid flow in continuous casting: (a) limiting case, no segregation, (b) negative segregation in central regions, (c) positive segregation in central regions.

more complete solutions have been used to model the formation of channel segregates. The models have recently been reviewed by Beckerman and Wang²⁴⁾ and by Prescott and Incropera.²⁵⁾ The more sophisticated (and therefore more mathematically intensive) models have thus far been applied only to limited extent to actual steels.

Fujii *et al.*²⁶⁾ as early as 1979 modeled low alloy steel macrosegregation taking account of the multicomponent nature of the alloy, and assuming either no diffusion or complete diffusion in the solid of each solute. He considered effects and the critical condition for channel formation (Eq. (5)). Subsequent notable papers on the subject are those of Ohnaka and Kobayashi²⁸⁾ and Bohmer *et al.*²⁹⁾

Fig. 10. Effect of bulging on interdendritic flow in vertically continuous cast carbon steel (Miyazawa and Scherdtfeger).³⁰⁾ (a) Schematic model of half thickness of continuous casting. (b) Left is flow assuming only bulging; right is flow including effects of both bulging and solidification shrinkage.

Poirier and Andrews²⁷⁾ extended the model to include porosity formation and predicted solute enrichment in upper portions of the casting and negative segregation near the bottom. Recent models which show the strong influence of the buoyancy effect on macrosegregation of steels are the 1999 papers of Gu and Beckerman³⁰⁾ and of Radovic *et al.*³¹⁾

Miyazawa and Schwerdtfeger³²⁾ were the first to demonstrate by quantitative calculation the effect of bulging on macrosegregation in continuous casting. Figure 10(a) illus-

Fig. 11. Macrosegregation of carbon (left) and of manganese (right) for three different degrees of bulging in carbon steel.³²⁾

trates their model, and Fig. 10(b) shows the liquid–solid zone. Resulting carbon and manganese macrosegregation is shown in Fig. 11. A method of considerable practical importance used to eliminate bulging is the application of external force and, preferably, introduction of a small amount of reduction (“soft reduction”) to reduce further undesirable fluid flow. Work of Ayata *et al.*³¹⁾ is one example. Continuous forging can also be employed, with similar resulting reduction of centerline segregation.³⁴⁾ Even in thin strip casting, some reduction during solidification improves homogeneity.³⁵⁾ Of course, care must be taken not to introduce too much deformation as severe segregation can then ensue as a result of solid movement as discussed later in this paper.

Some Things We Do Not Yet Fully Understand

There is work yet to be done in refinement of the overall mathematical model for macrosegregation and in developing accurate thermophysical properties needed for macrosegregation calculations. In addition there are still aspects of the basic segregation mechanisms we do not understand well. For example, we do not fully understand the extent to which convection in the bulk liquid can affect segregation in the semi-solid zone by directly influencing interdendritic flow or by carrying away floating crystallites. More important, perhaps, are other question relating to solid movement.

It is quite clear that settling of solid grains can occur in steel during the early stages of solidification. For example, it is well recognized that in a horizontally cast steel slab the columnar structure extending down from the top tends to extend further than the columnar zone growing upwards (when a central equiaxed zone also exists).³⁶⁾ What is not clear is the extent to which that settling affects macrosegregation. It seems to be widely accepted in the literature today that settling is the primary cause of the negative zone

of segregation in ingots. However, this has been questioned by a number of researchers^{6,7)} and it has been reported in the literature,⁶⁾ that a negative cone of segregation can be found in ingots that are fully columnar in the zone in question. As discussed earlier in this paper, a negative cone of segregation can be easily explained, at least qualitatively, on the basis of the expected interdendritic flow pattern without invoking an effect of crystallite settling.

When solid fraction is high enough that independent crystallite settling can no longer occur, a period of “mass flow” can occur during which the two phases, solid and liquid, flow together.^{6,12)} Such flow can easily be observed in wide freezing range alloys solidifying in relatively insulating molds. The top surface of the “riser” (“hot top”) settles uniformly downwards until a substantial fraction of solid is reached within the riser. Such movement of solid plus liquid also occurs in the central portion of steel castings and ingots, driven by metallostatic pressure, atmospheric pressure, and thermal or mechanical stresses.

The movement of the solid plus liquid can be homogeneous up to a certain fraction solid. Thereafter, local segregates (“filled hot tears”) form as a result of dilatancy along shear planes.³⁷⁾ This effect of shear on the weak semi-solid structure appears to be the primary cause of “V” segregates. The “V” segregates, however, may be expected to be enhanced by preferred interdendritic flow along the fissures opened.

It is understood that the mass feeding stage can be extended by decreasing grain size, the limit being reached in the semi-solid forming process in which a fine non-dendritic grain size is obtained;³⁸⁾ in this case homogeneous two-phase (solid plus liquid) flow can be obtained at solid contents over 50%. Thus, improved mass feeding may be the main reason why increasing the amount of equiaxed structure and decreasing grain size reduce macrosegregation.^{39,40)} Recently Nakada *et al.* have shown that application of a DC magnetic field during solidification of the center of continuous cast ingots reduced “V” segregates and macrosegregation.⁴⁰⁾ Presumably the magnetic field either enhanced the homogeneous mass feeding, or reduced the subsequent preferred interdendritic flow through fissures forming from the solid movement.

The movement of solid at high fractions solid provides a ripe and challenging area for future research on macrosegregation in steel ingots and continuous castings.

Conclusions

While there is much left to do to fully understand and control macrosegregation in practice, it can safely be said that in today’s casting and ingot making processes all types of macrosegregation form within the liquid–solid zone, not in front of it. In most cases, it is the result of slow interdendritic flow, driven by shrinkage, geometry, solid deformation or gravity. In some cases it may be the result of solid movement in the early stages of solidification. (*i.e.*, settling).

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