

E. FRAŚ\*, E. OLEJNIK\*

## INTERACTION BETWEEN SOLIDIFICATION FRONT AND ALIEN PHASE PARTICLES

### ODDZIAŁYWANIE CZĄSTEK FAZY OBCEJ Z FRONTEM KRYSZALIZACJI

Studies were carried out on two composites, i.e. succinonitrile – polystyrene using polystyrene particles of the radii R: 3, 6, 10, 15, 50, 100 and 200  $\mu\text{m}$  (K1) and succinonitrile – glass with glass particles of the radii R: 5.4, 12.0, 24.0, 38.9, 41.0 and 49.0  $\mu\text{m}$  (K2).

The directional solidification of the examined composite was carried out on a device for visualisation of the solidification process. The device is operating in a horizontal arrangement and uses a gradient temperature field in the space between the thermal elements. The device ensures stepless control of the sample movement velocity in a range from 0.01 to 100  $\mu\text{m}/\text{s}$ . From the studies carried out it follows that there is a critical velocity  $u_{\text{cr}}$  of the solidification front movement below which the particles are engulfed, and above which they are pushed. In other words, at the critical velocity there is a change of the active solidification front into a neutral one.

The critical velocity was determined for all radii R of the particles of the examined composite. It has been proved experimentally that the relationship between the particle radius and the critical front movement velocity is of a parabolic character  $u_{\text{cr}} = A/R$  where: A is complex composite material constant. From the regression analysis it follows that its value is  $A = 7.06 \mu\text{m}^2/\text{s}$  for K1 composite and  $A = 3.55 \mu\text{m}^2/\text{s}$  for K2 composite. To develop theoretical backgrounds for the phenomenon of the particle/solidification front interaction, the forces acting on a particle have been examined, i.e. the force of hydrostatic lift F, the drag force  $F_{\eta}$  caused by the viscosity of the liquid flowing around the particle, and a force caused by the surface tension  $F_{\sigma}$ . It has been proved that, using a balance of forces acting in the examined system, one can derive an equation for the critical front movement velocity  $u_{\text{cr}}$ , in the form of  $u_{\text{cr}} = a_0 \Delta\sigma / (3 R \alpha \eta)$ , where:  $a_0$  – interatomic distance,  $\Delta\sigma_0 = \sigma_{\text{p,s}} \cdot \sigma_{\text{p,l}}$ ;  $\sigma_{\text{p,s}}$  i  $\sigma_{\text{p,l}}$  – surface tension at the solid matrix – particle and liquid matrix – particle interface, respectively,  $\alpha$  – dimensionless coefficient,  $\eta$  – absolute viscosity of the liquid.

*Keywords:* solidification front, pushing and engulfment of particles, composites, succinonitrile- polystyrene and succinonitrile – glass system

Badaniami objęto dwa kompozyty, to jest nityl kwasu bursztynowego – polistyren (K1) z wykorzystaniem cząstek polistyrenu o promieniach: 3, 6, 10, 15, 50, 100 i 200  $\mu\text{m}$  oraz nityl kwasu bursztynowego – szkło z cząstkami szkła (K2): 5.4, 12.0, 24.0, 38.9, 41.0 i 49.0  $\mu\text{m}$ . Krystalizację kierunkową badanego kompozytu prowadzono w urządzeniu do wizualizacji procesów krystalizacji, działającym w układzie poziomym z gradientowym polem temperatury w przestrzeni pomiędzy elementami termicznymi. Urządzenie zapewnia płynną regulację prędkości przemieszczania próbki w zakresie od 0,01 do 100  $\mu\text{m}/\text{s}$ . Z przeprowadzonych badań wynika, że istnieje krytyczna prędkość  $u_{\text{cr}}$  przemieszczania frontu krystalizacji, poniżej której cząstki są wchłaniane a powyżej – odpychane przez front krystalizacji. Prędkość krytyczną wyznaczono dla wszystkich promieni R cząstek badanego kompozytu. Wykazano doświadczalnie, że zależność między promieniem cząstki a krytyczną prędkością przemieszczania frontu ma charakter hiperboliczny  $u_{\text{cr}} = A/R$  gdzie: A – kompleksowa stała materiałowa kompozytu. Analiza regresyjna wykazała, że jej wartość wynosi  $A = 7,06 \mu\text{m}^2/\text{s}$  dla kompozytu K1 oraz  $A = 3,55 \mu\text{m}^2/\text{s}$  dla kompozytu K2.

Na podstawie bilansu sił, działających w rozpatrywanym układzie, wyprowadzono równanie na krytyczną prędkość przemieszczania frontu  $u_{\text{cr}}$ , w postaci  $u_{\text{cr}} = a_0 \Delta\sigma / (3 R \alpha \eta)$ , gdzie:  $a_0$  – odległość międzyatomowa,  $\Delta\sigma_0 = \sigma_{\text{p,s}} \cdot \sigma_{\text{p,l}}$ ;  $\sigma_{\text{p,s}}$  i  $\sigma_{\text{p,l}}$  – napięcia powierzchniowe odpowiednio granicy stała osnowa – cząstka i ciekła osnowa – cząstka,  $\alpha$  – bezwymiarowy współczynnik przewodnictwa,  $\eta$  – lepkość dynamiczna cieczy.

\* AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY, FACULTY OF FOUNDRY ENGINEERING 23 REYMONTA AVN., 30-059 KRAKÓW, POLAND

## 1. Introduction

The effect of particles interaction with solidification front is observed in a variety of phenomena, to mention just the composites cast *ex situ* and *in situ*, the segregation of inclusions in castings, the growth of monotectics, the zone refining of metals, and some fields of cryobiology (storage of biological cells in crystallising solutions of salts). Depending on the type of the solidification process, we can effectively utilise either the active role of the solidification front which pushes the particles of an alien phase, or the neutral role which results in their engulfment. Figure 1 shows schematically both active and neutral role of the front during directional solidification and solidification in volume.

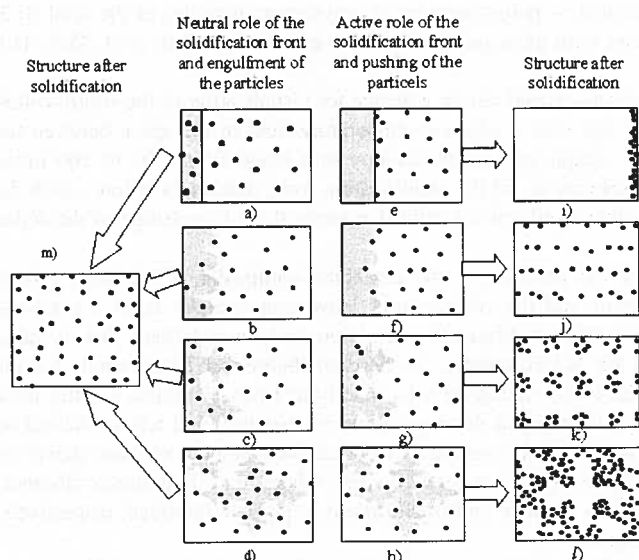


Fig. 1. Active/neutral role of the solidification front during its interaction with particles of an alien phase and the resulting structural effects; the solidification front during directional solidification: (a) planar, (b) cellular, (c) cellular-dendritic, (d) solidification in volume, (e), (i)÷(l) structure finally obtained

In the case of engulfment (Fig. 1a÷1d), the particles of inclusions are carried to the crystal interior and are homogeneously distributed there (Fig. 1m). When particles are pushed by the solidification front (Fig. 1e÷1h), they gather in the remaining portion of the liquid metal and are moving at the same velocity as the velocity of the advancing front. As the process of solidification progresses, the pushed particles are engulfed by the moving phase boundaries. During directional solidification with a planar front, the pushed particles are transferred to the far end of an ingot (Fig. 1i). When the particles are pushed by a cellular solidification front, they gather in the intercellular spaces and are next enclosed mechanically by the dendritic cell boundaries (Fig. 1j). During directional solidification with a cellular-dendritic front, the pushed particles are enclosed mechanically in the

spaces between the dendritic cells (Fig. 1k). On the other hand, the solidification in volume makes them enclosed mechanically in spaces between the dendrite branches (Fig. 1l). Hence a conclusion follows that pushing or engulfment of the particles is the main cause of heterogeneities observed in the structure of materials. The type of the heterogeneity (Fig. 1i÷1l) depends on the shape of the solidification front.

The type of an interaction that occurs between the alien phase and the solidification front is very important. For example, to improve the mechanical properties of materials, the process of their solidification should proceed in a way such as to ensure a homogeneous distribution of particles (Fig. 1m) and avoid the formation of clusters which will act as notches. In other words, the solidification front should engulf the particles, which means that it should be neutral in respect of them. The requirements are quite opposite when high-quality monocrystals are to be produced or when the solidifying material is to be zone-refined. In such cases the solidification front should be active in respect of the particles of alien phases, which means that it should push them away. Due to this, the undesired inclusions will gather on one side of the crystal (Fig. 1i), and when this part of the crystal is removed, the remaining material will be free from any inclusions.

The authors of some studies tend to associate the mere fact of finding and identifying an alien inclusion in a crystal with finding and identifying a nucleus of this crystal, which leads to quite false conclusions [1]. In reality [2], the fact that an alien inclusion is present in a crystal (e.g. Fig. 2a) does not mean that it will act as a substrate for crystal nucleation. It is possible that the inclusion has been engulfed "mechanically" in the crystal, while the nucleus of this crystal has been formed in a place different from that of an inclusion. This is best proved by a photo in Figure 2b, which shows several inclusions of TiC in the same dendritic crystal. Since it is well-known [4] that one single crystal is formed from one single nucleus, it certainly cannot be the inclusion of TiC.

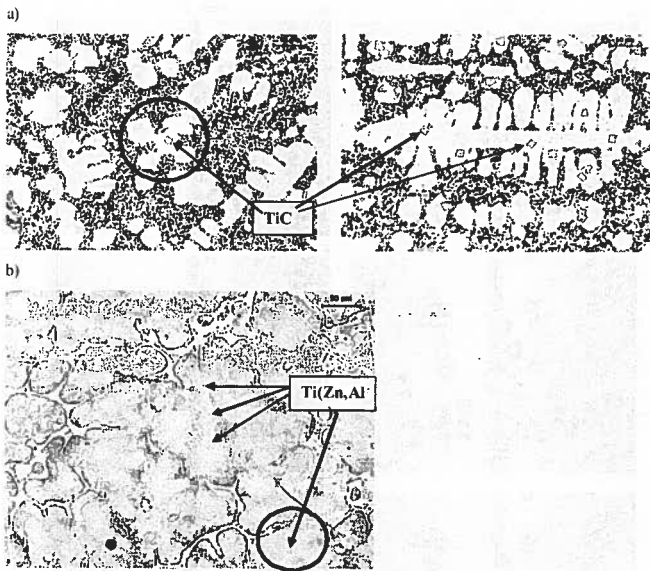


Fig. 2. Single TiC and Ti(Zn,Al) particles visible on the cross-section of dendrite and numerous particles of TiC and Ti(Zn,Al) visible on the lengthwise section of the dendrite; a) cast iron, b) Zn-25%Al-4%Ti alloys

## 2. Methods of experimental research

Studies were carried out on two composites, i.e. succinonitrile – polystyrene using polystyrene particles of the radii  $R$ : 3, 6, 10, 15, 50, 100 ( $K_1$ ) and succinonitrile – glass with glass particles of the radii  $R$ : 5.4, 12.0, 24.0, 38.9, 41.0 and 49.0  $\mu\text{m}$  ( $K_2$ ). The composite matrix (succinonitrile) is a transparent material of low melting point. Its solidification is quite easy to observe under an optical microscope. Due to its crystalline structure and the well-known values of the entropy changing during solidification it makes a perfect material for simulation of the solidification process proceeding in metals. To obtain a directional solidification in the examined composites, a special device was designed. It is operating in a horizontal system and ensures visualisation of the solidification process. The device is schematically depicted in Figure 3. The examined sample of transparent organic material (10) is placed on work stage (2) of microscope (1) between heating (2a) and cooling (2b) elements with adjustable distance. The heating elements were made by serigraphy on ceramic plates. The cooling elements are heat pumps (Peltier cells), cooled on the hot side with water of temperature stabilised by system (9). The thermal elements are mounted on the stage frame with option to change the distance between them. The specimen is moving across the gradient field of temperature and within a space between the thermal elements following the direction of temperature gradient. The screw of the specimen feed system (3) is connected to gear (4), enabling

stepless adjustment of the specimen feed rate within a range from 0,01 to 100  $\mu\text{m/s}$ .

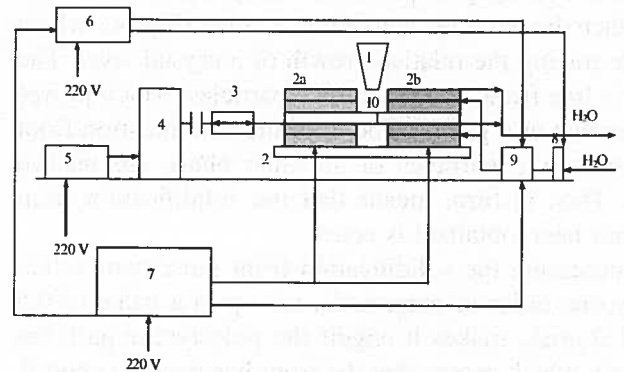


Fig. 3. Schematic representation of a stand for visualisation of the solidification process

## 3. The results

The photos in Figures 4-9 show examples of a sequence of the solidification front-particle interaction recorded for different front movement velocities and particles radii.

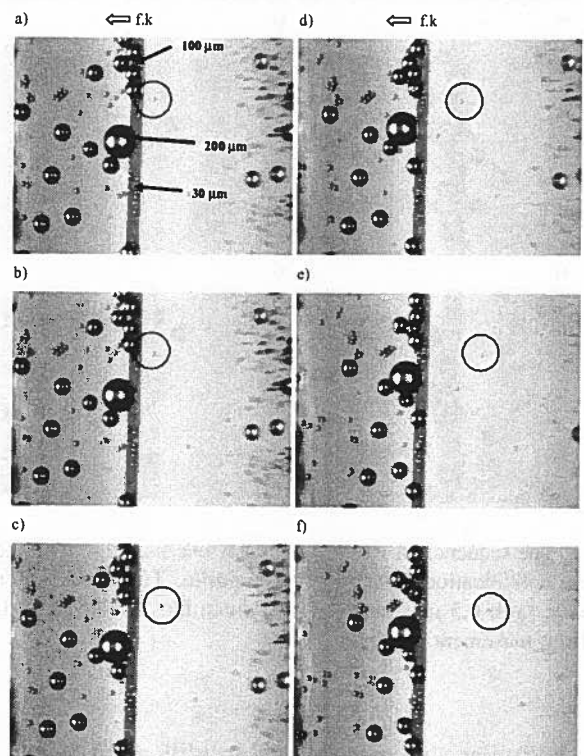


Fig. 4. The sequence in which the polystyrene particles are pushed by the solidification front in  $K_1$  composite. The front movement velocity (a,b) 0.05  $\mu\text{m/s}$ ; (c,d) 0.07  $\mu\text{m/s}$ ; (e,f) 0.08  $\mu\text{m/s}$ . f.k – solidification front,  $\leftarrow$  front movement direction

From photos in Figure 4 it follows that planar solidification front advancing at a rate of 0.05, 0.07 and 0.08  $\mu\text{m/s}$  pushes the polystyrene particles of radii 15, 50 and 100  $\mu\text{m}$ . It is easy to prove this adopting as a point of reference the particles marked by a circle (Fig. 4), which enable tracing the relative growth of a crystal layer. The layer is free from the polystyrene particles, which proves further that at a given velocity of the solidification front movement the particles of an alien phase are pushed away. This, in turn, means that the solidification front that has been obtained is active.

Increasing the solidification front movement velocity by one order of magnitude, i.e. up to a value of 0.5 and 1.0  $\mu\text{m/s}$ , makes it engulf the polystyrene particles (Fig. 5), which means that the front has become neutral.

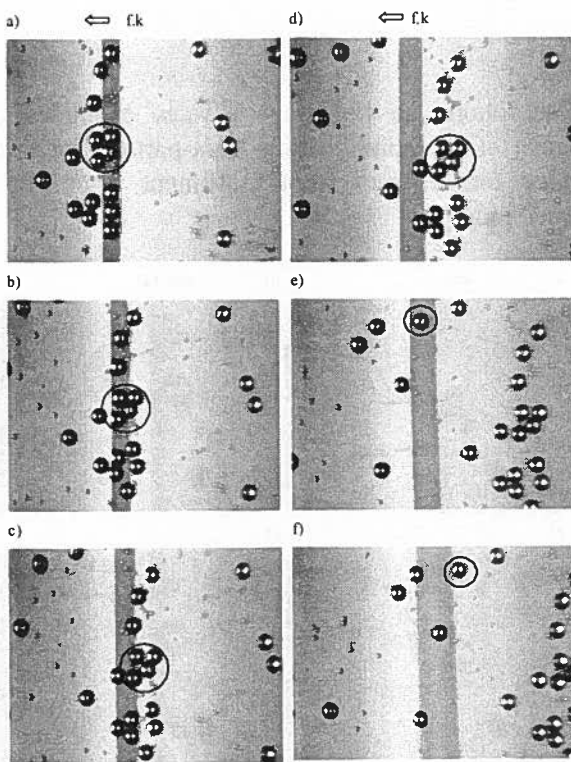


Fig. 5. The sequence in which the polystyrene particles are engulfed by the solidification front in K1 composite. The front movement velocity: (a-d) 0.5  $\mu\text{m/s}$  and (e,f) 1.0  $\mu\text{m/s}$ ; f.k.- solidification front,  $\leftarrow$  front movement direction

A similar sequence of the solidification events has been recorded for the composite with glass particles. With the solidification front movement velocity amounting to 0.01 and 0.03  $\mu\text{m/s}$ , the glass particles are pushed by the solidification front (Fig. 6), while at higher velocities of 0.5 and 1.0  $\mu\text{m/s}$ , the front tends to engulf them (Fig. 7).

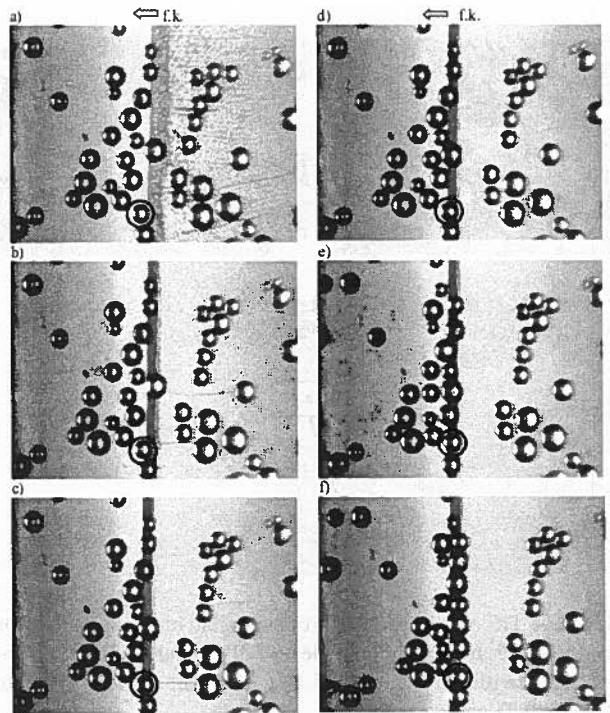


Fig. 6. The sequence in which the glass particles are pushed by the solidification front in K2 composite. The front movement velocity: (a) - (e) 0.01  $\mu\text{m/s}$  and (f) 0.03  $\mu\text{m/s}$

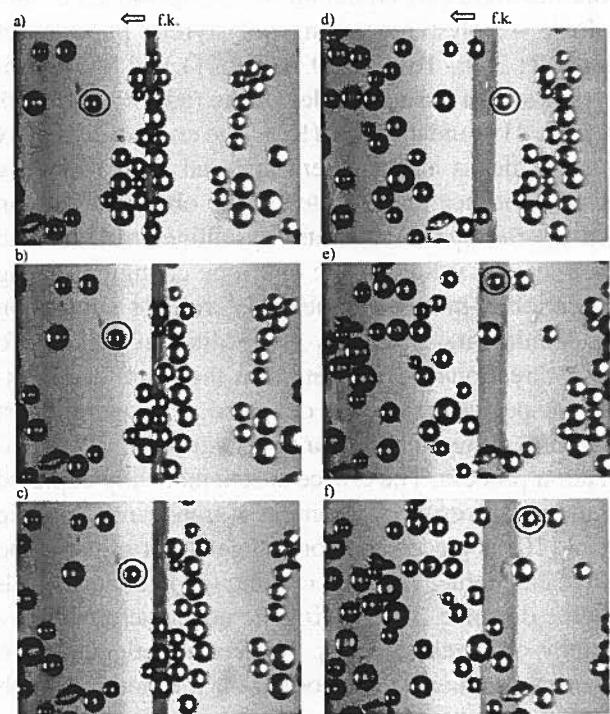


Fig. 7. The sequence in which the glass particles are engulfed by the solidification front in K2 composite. The front movement velocity: (a) - (d) 0.5  $\mu\text{m/s}$ , (e), (f) 1.0  $\mu\text{m/s}$

Figure 8 shows the sequence of changes in the shape of the solidification front in the vicinity of a polystyrene



particle in composite K1 in function of the front movement velocity. From the drawing it follows that the shape of the steady front is convex (Fig. 8a). When the front starts advancing at a velocity of  $0.1 \mu\text{m/s}$ , its shape changes from convex into planar (Fig. 8b); the particle is pushed. With the velocity raised to  $0.15 \mu\text{m/s}$ , the shape of the solidification front changes to concave and the particle continues being pushed (Fig. 8c-f). A general observation is that in composite K1 changes in the solidification front migration velocity in a range from 0 to  $0.15 \mu\text{m/s}$  make the front change its shape from convex to concave.

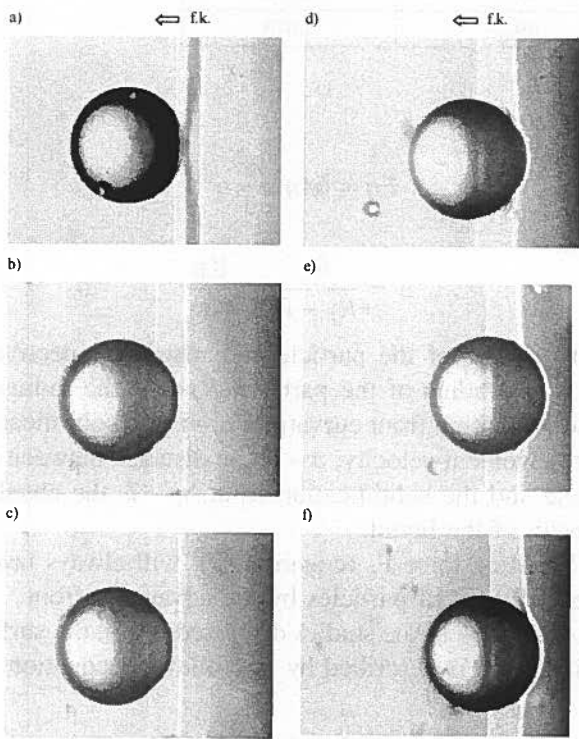


Fig. 8. The effect of growth rate on the shape of the solidification front in the vicinity of a polystyrene particle in K1 composite; K1; (a)  $u = 0.00 \mu\text{m/s}$ , (b)  $u = 0.10 \mu\text{m/s}$ , (c)  $u = 0.15 \mu\text{m/s}$  after 5 min, (d)  $u = 0.15 \mu\text{m/s}$  after 15 min, (e)  $u = 0.15 \mu\text{m/s}$  after 25 min, (f)  $u = 0.15 \mu\text{m/s}$  after 35 min – the state of equilibrium (the time was counted since the moment when the solidification front movement velocity changed). The size of particles on the front was  $200 \mu\text{m}$

Like in previous case, also composite K2 has been observed to change the shape of its solidification front (Fig. 9).

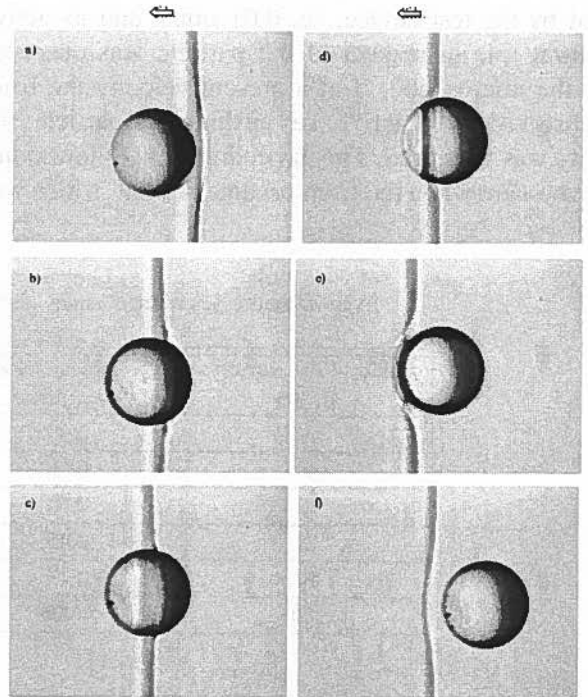


Fig. 9. The sequence in which the glass particles are engulfed by the solidification front in K2 composite. The front movement velocity was (a)  $0.01 \mu\text{m/s}$  during pushing and (b) – (f)  $0.035 \mu\text{m/s}$  during engulfment of the particles, respectively

#### 4. Discussion of results

From the results obtained in the research it can be concluded that there is a critical velocity  $u_{cr}$  of the solidification front movement below which the particles are engulfed, and above which they are pushed. In other words, at a critical velocity, the solidification front changes from active into neutral.

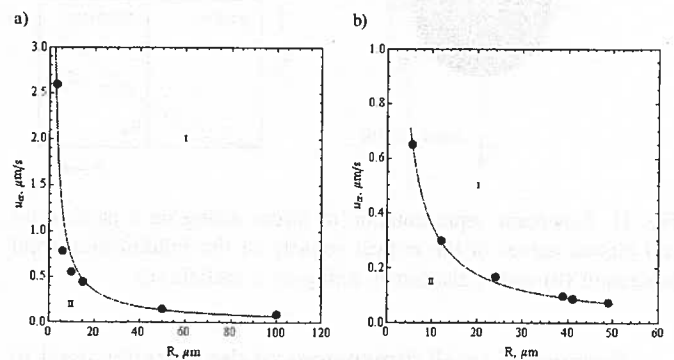


Fig. 10. The effect of the particle radius  $R$  on the critical velocity  $u_{cr}$ , I – area of particle engulfment, II – area of particle pushing, (a) K1 composite, (b) K2 composite

To determine the critical velocity for a composite with particles of a given size, the solidification front was moved, selecting first the lowest possible velocity

offered by the test device, i.e.  $0.01 \mu\text{m/s}$ , and its active or neutral role in respect of the particle was observed under the microscope. If at a present velocity the front was observed to be active, i.e. pushing the particle, the velocity was increased. This procedure was followed until the moment when the front became neutral, which was

revealed in an engulfment of the particles. The velocity at which the particles are engulfed by the front has been called the critical velocity  $u_{cr}$ . The critical velocity was determined for all radii  $R$  of the particles of the examined composites. The results of the studies are compiled in Table 1 and depicted in Figure 10.

TABLE 1

Experimentally determined values of the critical velocity for K1 and K2 composites

Radii of the particles $R, \mu\text{m}$	The critical velocity, $u_{cr}, \mu\text{m/s}$	Radii of the particles $R, \mu\text{m}$	The critical velocity, $u_{cr}, \mu\text{m/s}$
3	2.60	5.4	0.650
6	0.78	12.0	0.300
10	0.56	24.0	0.170
15	0.45	38.9	0.097
50	0.15	41.0	0.086
100	0.096	49.0	0.073

To explain the effect of particles interaction with solidification front, the forces acting onto these particles have been examined. From [4] it follows that on the particle of a radius  $R$  immersed in liquid and placed at a distance  $d$  from the solidification front, three forces are acting (Fig. 11a), viz. the buoyant force  $F$ , the drag force  $F_\eta$  and the surface tension force  $F_\sigma$ .

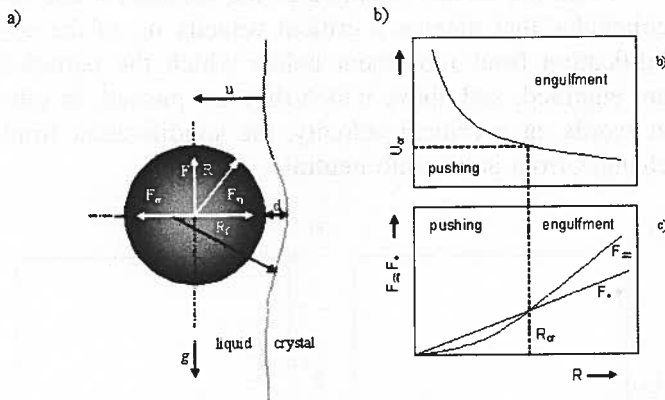


Fig. 11. Schematic representation of forces acting on a particle (a) and plotted curves of the critical velocity of the solidification front movement (b) and of the forces acting on a particle (c)

Because of small dimensions of the particles used in ours composites, the buoyant force  $F$  is negligibly small and as such can be disregarded in the overall balance of forces.

The drag force  $F_\eta$  is caused by the viscosity  $\eta$  of liquid flowing around the particle of a radius  $R$ , and as such it depends on the front curvature. According to the studies described in [3] it can be written in the following form:

$$F_\eta = 6\pi\eta u \frac{R^2}{d} \alpha^2 \quad (1)$$

where:

$$\alpha = \frac{R_f}{R_f - R} = \frac{K_p}{K_m} \quad (2)$$

$K_p, K_m$  – are of the particle and matrix, respectively;  $R$  – is the radius of the particle;  $R_f$  – is the radius of the solidification front curvature;  $u$  – is the solidification front movement velocity;  $d$  – is the distance between the particle and the solidification front,  $\eta$  – is the absolute viscosity of the liquid.

The drag force  $F_\eta$  (equation (1)) will always favour the engulfment of particles by the advancing front.

According to the studies disclosed in [4], the surface tension force is described by the following equation:

$$F_\sigma = 2\pi R \Delta\sigma \left(\frac{a_0}{d}\right)^2 \alpha \quad (3)$$

where:  $a_0$  – is the interatomic distance,  $\Delta\sigma = \sigma_{p,s} - \sigma_{p,l}$ , where  $\sigma_{p,s}, \sigma_{p,l}$  – are the surface tension at the particle-solid interface and particle-liquid interface, respectively.

For  $\Delta\sigma < 0$ , force  $F_\sigma$  (equation (3)) is acting in the same way as the resisting force and favours engulfment of particles by the moving front. When  $\Delta\sigma > 0$  (i.e. when the surface tension at the particle-liquid interface is lower than at the particle-solid interface), this force will favour pushing of particles by the solidification front. Let us consider now the case when force  $F_\sigma$  is the pushing force and is compensated by the drag force  $F_\eta$  at a constant velocity  $u$  of the solidification front movement. The equilibrium of forces keeps the particle at a constant

distance  $d$  from the solidification front (Fig. 11a). Under such conditions, the velocity of the particle and of the solidification front movement will be the same. This state will persist until the moment when the front movement velocity increases, and then, according to equation (1), the resisting force  $F_\eta$  will increase, too. The force  $F_\sigma$ , caused by the surface tension will not change, since its value does not depend on the velocity  $u$ . This means that the state of the mechanical pseudo-equilibrium, when the particle is pushed by the front, has been disturbed. As a consequence, the drag force  $F_\eta$ , will be higher, ultimately resulting in a contact between this particle and the solidification front, followed by engulfment of the particle. From this statement it follows that there is a critical value of the velocity  $u_{cr}$ , of the front movement which, when exceeded, will make the particle engulfed by the solidification front. An analytical determination of this critical velocity is based on the balance of forces acting in the examined system (Fig. 11a)

$$F_\sigma - F_\eta = 0 \quad (4)$$

Having substituted equations (1) and (3) to (4)

$$2\pi R \Delta\sigma \left(\frac{a_0}{d}\right)^2 \alpha - 6\pi\eta u \frac{R^2}{d} \alpha^2 = 0 \quad (5)$$

it is possible to determine the critical velocity of the solidification front movement

$$u_{cr} = \frac{a_0^2 \Delta\sigma}{3dR\alpha\eta} \quad (6)$$

Assuming that the smallest distance between the particle and the solidification front is equal to an atomic diameter  $d = a_0$ , the equation for the critical velocity  $u_{cr}$  can be written as

$$u_{cr} = \frac{a_0 \Delta\sigma}{3R\alpha\eta} = \frac{A}{R} \quad (7)$$

where:  $A$  – is the complex material constant of the composite.

From equation (7) it follows that the relationship between the particle radius and the critical velocity of the front movement is of a hyperbolic character (Fig. 11b). Figure 11c shows a relationship between the radius  $R$  of a particle and the magnitude of forces  $F_\eta$  and  $F_\sigma$  for constant velocity  $u$  of the solidification front movement to which is corresponding the critical radius of a particle  $R_f$ . For particles of a radius  $R > R_f$  the drag force  $F_\eta$  “glueing” the particle to the front is prevailing over the pushing force  $F_\sigma$ , which results in an engulfment of particles by the solidification front. Hence it can be concluded that the solidification front is neutral in respect of the particle.

In the opposite case, when  $R < R_f$  the force  $F_\sigma$  is prevailing and the particle is pushed by the solidification front; the front becomes active in respect of the particle which results in the formation of clusters of particles and, as a consequence, in structural heterogeneities.

The relationship between the radius of a particle and the critical velocity as expressed by equation (7) has been confirmed by the authors' own investigations, graphically depicted in Figure 10. Having allowed for the data compiled in Table 1 and for equation (7), the value of the material constant  $A$  can be determined from an analysis of regression. The value of this constant for a succinonitrile – polystyrene composite is  $A = 7.06 \mu\text{m}^2/\text{s}$ , while for a succinonitrile – glass composite it is  $A = 3.55 \mu\text{m}^2/\text{s}$ . For both composites the coefficient of correlation is high and exceeds the value of 0.97.

From the hypothesis put forward in [5] it follows that the effect of pushing or engulfment of particles by the solidification front depends on the value of the dimensionless coefficient  $\alpha$ ; where for  $\alpha < 1$ , the particles are pushed, while for  $\alpha > 1$ , the particles are engulfed by the advancing solidification front. The values of the dimensionless coefficient for composites K1 and K2 are  $\alpha = 0.35$  and  $\alpha = 5.63$ , respectively. The conducted experiments have not been able to prove the basic assumption made in this hypothesis, since in both cases, irrespective of a value of the coefficient, the particles were pushed and engulfed by the advancing solidification front.

## 5. Conclusions

1. It has been proved that, depending on the size of the particles and the velocity of the solidification front movement, the front can be either active or neutral in respect of the composite particles.
2. The active solidification front by pushing the particles favours the formation of clusters, which cause microstructural heterogeneities in the composite.
3. With neutral solidification front, the particles are engulfed to the crystal interior and, as a result of this effect, composites of a homogeneous microstructure are obtained.
4. The neutral/active change of solidification front in respect of the particles of a given size takes place at a critical velocity which forms a hyperbolic relationship with the particle size.

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...the effect of particle size on the critical velocity of the solidification front is shown in Figure 2. It can be seen that the critical velocity decreases as the particle size increases. This is due to the fact that larger particles have a larger surface area, which leads to a higher rate of heat transfer and a faster solidification front velocity.

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Figure 2. Critical velocity of the solidification front as a function of particle size.

$$v_{cr} = \frac{2 \cdot \sigma \cdot \gamma}{\rho \cdot \Delta T \cdot r_p} \quad (1)$$

where:  $v_{cr}$  - critical velocity of the solidification front, m/s;  $\sigma$  - surface energy, J/m<sup>2</sup>;  $\gamma$  - contact angle, degrees;  $\rho$  - density, kg/m<sup>3</sup>;  $\Delta T$  - supercooling, K;  $r_p$  - particle radius, m.

$$v_{cr} = \frac{2 \cdot \sigma \cdot \gamma}{\rho \cdot \Delta T \cdot r_p} \quad (2)$$

...the critical velocity of the solidification front is shown in Figure 2. It can be seen that the critical velocity decreases as the particle size increases. This is due to the fact that larger particles have a larger surface area, which leads to a higher rate of heat transfer and a faster solidification front velocity.

$$v_{cr} = \frac{2 \cdot \sigma \cdot \gamma}{\rho \cdot \Delta T \cdot r_p} \quad (3)$$

...the critical velocity of the solidification front is shown in Figure 2. It can be seen that the critical velocity decreases as the particle size increases. This is due to the fact that larger particles have a larger surface area, which leads to a higher rate of heat transfer and a faster solidification front velocity.

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