The Upper Temperature Limit Above which the Influence of Dislocations Due to the Presence of the Reinforcement of the Composite 6061/SiC becomes Negligible

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Abstract: ***Studies that have already been made, shows that the acceleration of the precipitation kinetics of the semi coherent phase is attributed to the dislocations created during tempering, due to the difference in thermal expansion coefficient of the reinforcement and the matrix. In this study, we seek to clarify this effect by trying to define the upper limit temperature above which the influence of dislocations due to the presence of the reinforcement becomes negligible.***

Keywords: Alumium, SiC, Composite, Dislocation, precipitation

1. Introduction

Analysis of experimental results discussed in the alloys 6061 reinforced by SiC aluminium composite (Dafir 2012), we found a generalized acceleration of the precipitation in the composite relative to that observed in the non-reinforced matrix and the all types of precipitation (precipitation coherent , semi- coherent and incoherent).This result , as we have seen in the bibliography, was already known in the case of semi- coherent precipitation for which the origin of the acceleration is attributed to dislocations created during quenching due the difference between the values of the coefficients of thermal expansion of the reinforcement and the matrix. In this study, we seek to clarify this effect by trying to define the upper temperature limit above which the influence of dislocations due to the presence of the reinforcement becomes negligible. As against, causing the acceleration of the subject remains coherent precipitation of discussion. Recall that it was ascribed to the presence of dislocations (increase of the overall contribution of the scatter distribution in heart) , and / or enrichment of the solute adjacent reinforcement , resulting from the existence of a gradient stresses in the material. Furthermore, to our knowledge, no studies have been carried out on the precipitation of an incoherent phase in a composite. It is however necessary to consider the effects may be observed in this case, in order to establish a comprehensive interpretation of the influence of reinforcement on precipitation. Finally, a preliminary analysis of the initial state brought to the annealing temperature (density of dislocations, internal stresses) material is also required before any attempt at interpretation.

2. Evolution of internal stresses and plastic deformation state of the material as a function of the tempering temperature

2.1. Dislocations

Matrix composite quenched contains a high density of dislocations; in a temperature maintenance can be eliminated through the restoration. Without a comprehensive study of the kinetics of this phenomenon each aging temperature, we tried to engage the qualitative characteristics with experience in situ electron microscopy. For this, we followed the removal of dislocations in a thin composite, in a slow rise in temperature, from ambient to 535°C, at a heating rate of about 10°C / minute.

Figure.1, shows some images from the video recording made during in-situ heating of the thin blade. At 125°C, there is observed in the vicinity of the reinforcement, with a very high density of dislocations. That persists at 200 ° C, and it is only at temperatures of about 300°C to 320°C, that there is a beginning of mobility of dislocations leads to a partial restoration of the material and then a gradual recovery in the range 300°C - 400°C. However, even at temperatures as high as 500°C, long dislocations remain anchored to the precipitates in the matrix.

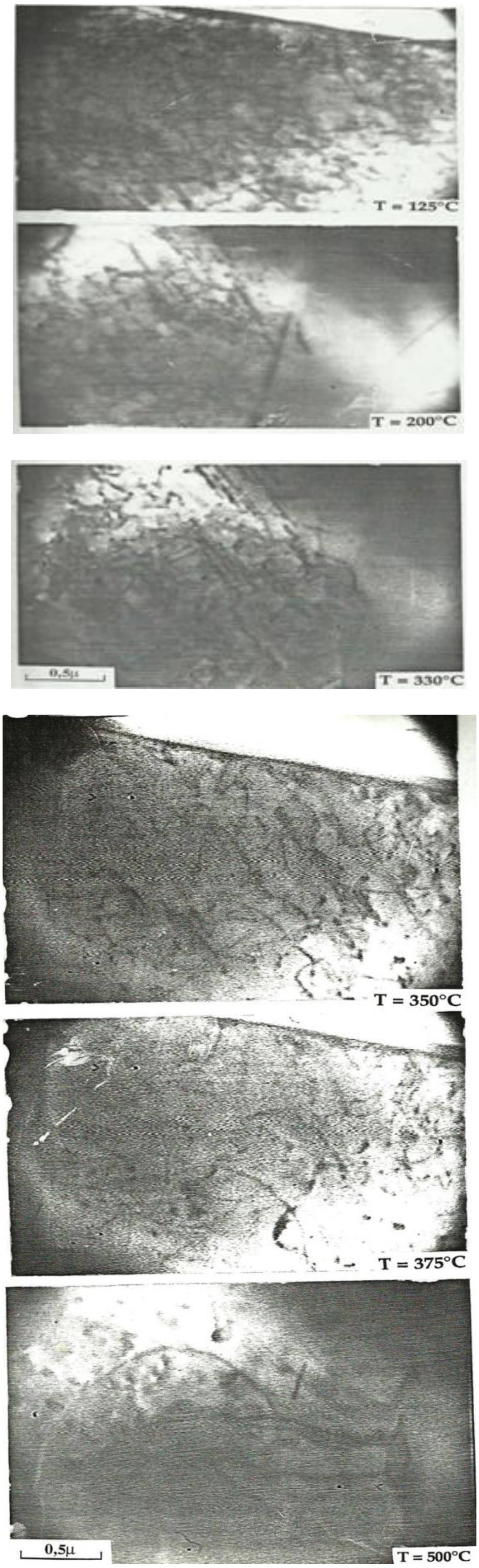

Figure 1: Images of video recording of a heated blade in situ

This experience allows us to draw the following conclusions:

In the area of precipitation of β " phase (125°C < T < 200°C), it is likely that the mobility of dislocations is very low, and the evolution of internal stresses during the implementation temperature, simply characterized, in the absence of a restoring material, and a lower field reversal elastic residual stress due to difference in thermal expansion coefficient between the reinforcement and the matrix. This stress state should remain substantially constant during the precipitation kinetics or at least for a time of the same order of magnitude as that required for the precipitation of the coherent phase. We refer to the precipitation kinetics, there is indeed at 200 ° C for example, the time precipitation of β'' phase is about 1 hour (Dafir 2012), whereas in our in situ experiment, the rise time to 300 ° C, at which temperature there was still a significant dislocation density is of the order of 30 minutes .

In the area of the precipitation step β " (230 ° C < T < 300 ° C), the restoration of the material becomes appreciable. However, the persistence of the dislocations in high temperature, allows supposing that, in the temperature range considered, the rate of restoration is enough that in the initial phase, very short, germination precipitates β ' should therefore be only dictated by the quenching conditions.

2.2 Internal Constraints

We wanted to assess the state of internal stresses in the material in the field of coherent precipitation β ". This evaluation was performed using two models:
a) The model of a spherical inclusion (Dafir1993) in an infinite matrix placed to determine:

- Configuration of the dislocation network (number, distance relative to the equilibrium particle) created during cooling.
- The field of residual stresses in the spring and in the particle matrix (Hamann 1992).

b) A purely elastic model that, based on an iterative method based on the theory of Eshelby, to determine the scope of residual thermal stress in a composite containing a fraction f of particles (Hamann 1992). The treated case is that of a composite 6061/SiC 20% volume fraction of particles. The calculations were made in plasticity and damage MATEIS group (INSA Lyon).

Figure.2 shows the qualitative change during cooling:

- The average frictional stress τ_f (network) in the sliding plane of the dislocation according to temperature (dashed curve). This curve is derived from the literature data (Hirsch and Warrigton 1961).
- This same constraint of friction, taking into account the issue of dislocations during tempering τ_f that contribute to an end by $\Delta\tau_d$. We assumed that restoration of the material is such that the dislocations in the matrix is subjected to a temperature below 350°C.

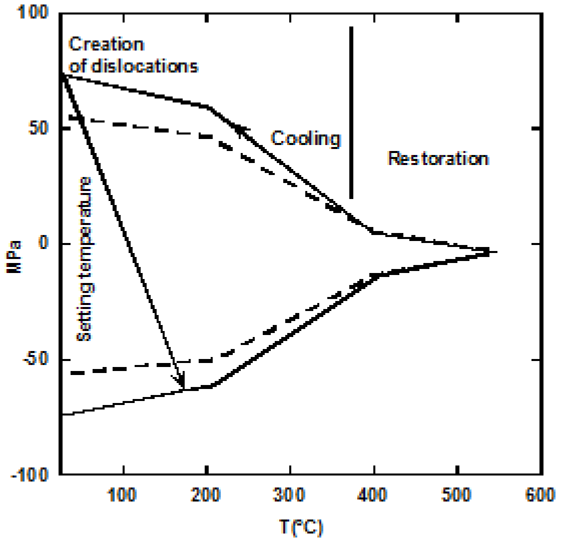

Figure 2: Evolution in function of the temperature of friction stress in a 6061 matrix in the vicinity of a spherical particle of SiC.

A hardened state, the model gives us a numerical value of the radial component of the tensor of elastic stresses in the particle of about 100 MPa and the total amplitude of the stress of friction is at room temperature, equal to the critical stress resolved in the slip plane, about 75 MPa. We also in the case of composite and a purely elastic deformation calculating Δ Teff leads to the same constraint in the particle. Figure.3 shows changes depending ΔT in the matrix. We see that a stress of 100 MPa in the inclusion is a ΔT_{eff} about 70°C. Field image corresponding < σ_{im} > in the composite is then approximately 24 MPa.

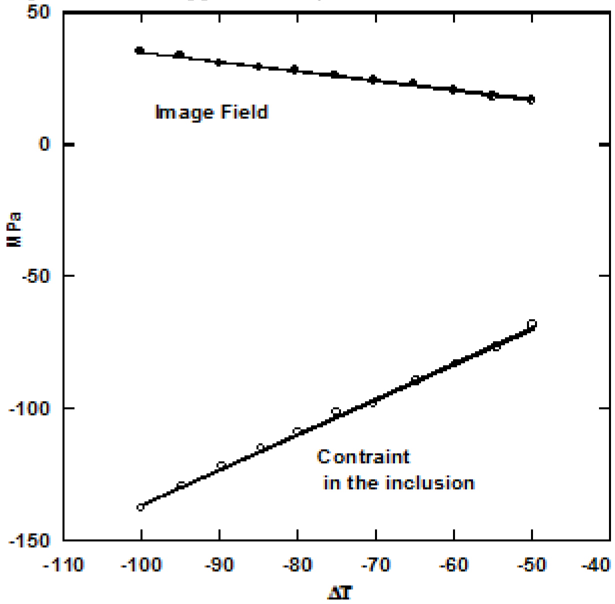

Figure 3: Stress in the inclusion and average image field in the composite 6061/20 SiCp generated by a change in temperature. DELTA.T. For a purely elastic accommodation. Calculation by the iterative model of Hamann

The radial and orthoradial σ_{rr} constraints $\sigma_{\Theta\Theta}$ matrix in the case of a material containing a volume fraction f of the particles can be approximated by adding the average value of the field profile by calculating the Eshelby method in the case of an infinite matrix (Figure.4 and figure.5). The radial stress is a compressive stress, while shear stress is typical voltage.

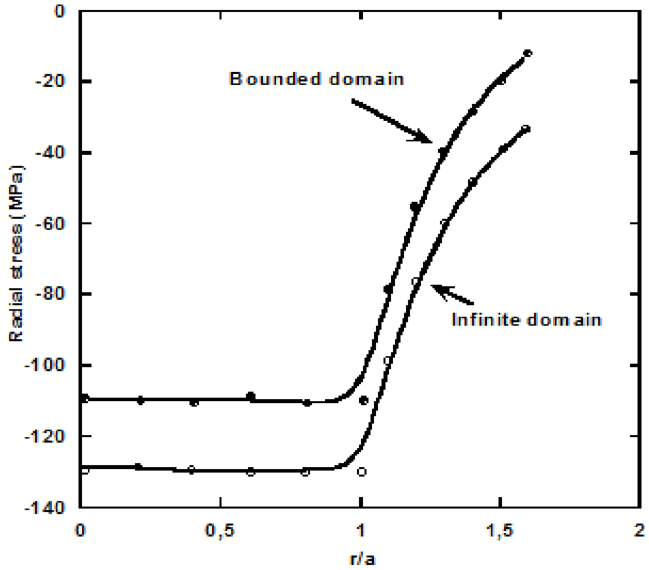

Figure 4: Radial Constraint

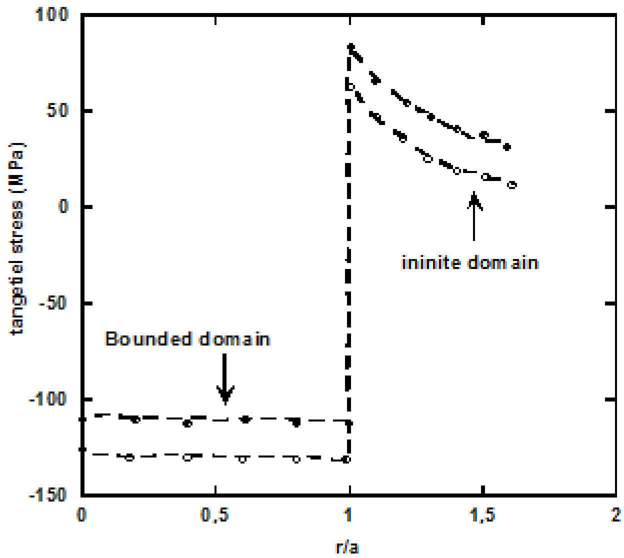

Figure 5: Tangential stress

The question is then to determine the stress state of the worn after quenching temperature aging material.

Presumably they remain in constant dislocation structure as during warming up, the driving stress τ_m dislocations remains between τ_f and $-\tau_f$. Under these conditions, if the composite is brought to temperature, is first entirely assists variation τ_m. According to the foregoing τ_f on the surface of the particle should be canceled to 100°C and then reverse to reach f about 170 ° C. Figure.1 shows schematically such a development.

The elastic constraint in the inclusion ranges from about 70 MPa (Fgure.3) when the temperature varies from 50°C which corresponds to a variation in stress determined the sliding plane of approximately 50 MPa. An increase in temperature of about 140°C would be necessary to obtain, dislocation structure constant, a complete reversal of this constraint resolved in the sliding plane in the vicinity of the particle, which makes us to a temperature of 170°C , in the range of raining β ".

3. Influences of different microstructural parameters on the precipitation

3.1 Influence of dislocations

Outside the role of preferential nucleation site for heterogeneous precipitation, the effect of diffusion in the heart associated with dislocations sometimes has been suggested to explain the acceleration of precipitation kinetics consistent (Dutta et al 1991). Moreover, the hypothesis of a reduction of the free energy of formation of precipitates due to the interaction of the dislocation strain fields and precipitates was also proposed to explain this phenomenon.

To verify these hypotheses, we experimentally studied the kinetics of precipitation PTE to 125 ° C and 175 ° C of 6061 hardened to 4% after quenching and non- hardened (Figure .6 and.Figure.7). We note that the curves PTE present a look identical, so that the hardening has no significant effect on the kinetics of precipitation of the coherent phase. The only difference observed is that a level of PTE slightly lower in the work-hardened samples (especially in the case of the kinetics at 175 ° C). This can be attributed to a slight initial decrease effect drainage solute by dislocations (Cottrell atmosphere) during hardening (Merle et al 79).

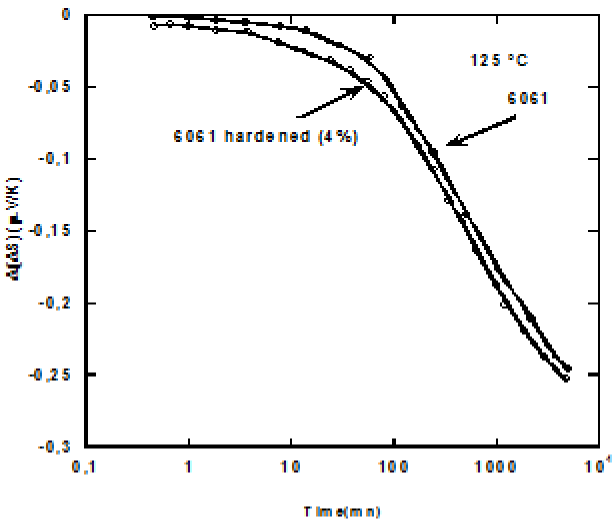

Figure 6: Study of the influence of dislocations on the precipitation kinetics of the β 'phase' .T = 125 ° C

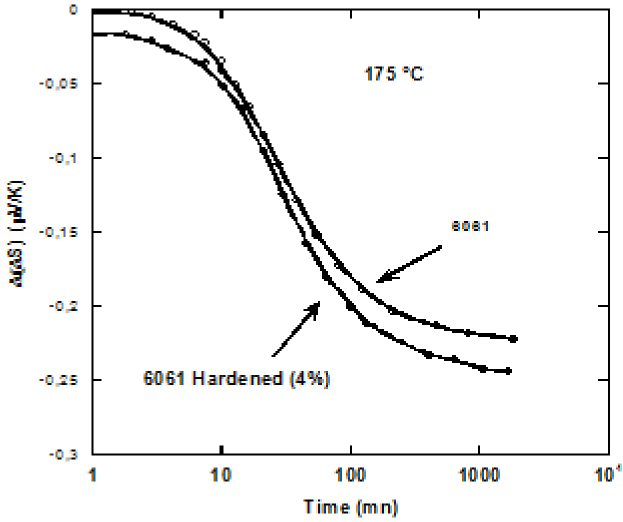

Figure 7: Study of the influence of dislocations on the precipitation kinetics of the β 'phase' .T = 175 ° C.

3.2 Influence of the stress field

The theoretical evaluation presented (Dafir 2012) shows us, in the temperature range where we studied the precipitation β'', we should see significant of the stress state of the material. According to estimates, the stress field in the matrix effect should be reversed to a temperature region of 100 ° C and increase to 100°C and 175°C. However, we found experimentally that the acceleration of the coherent precipitation rate also remained essentially constant in the field of temperature (Massardier 91). The comparison between our experiments and calculations, we therefore doubt the existence of a strong influence of the field of internal stress on the precipitation, at least in the case of this alloy.

To verify this conclusion, we attempted to vary artificially the state of stress in the material and provide an experimental answer to the following two questions:

1) A state of uniform stress he has an influence on the coherent precipitation?
2) A change in the stress gradients in the reinforced material he also influenced?

To do this we compared the kinetics of aging at 125°C and 175°C a sample of the unreinforced matrix , unconstrained or subjected to a uniform stress $2\sigma_E / 3$, σ_E is the elastic limit at this temperature (σ_E = 70 MPa).

These samples were aged simultaneously to eliminate any difference in heat treatment. Perfect identity between the two kinetics is observed (Figure.8 and Figure.9).

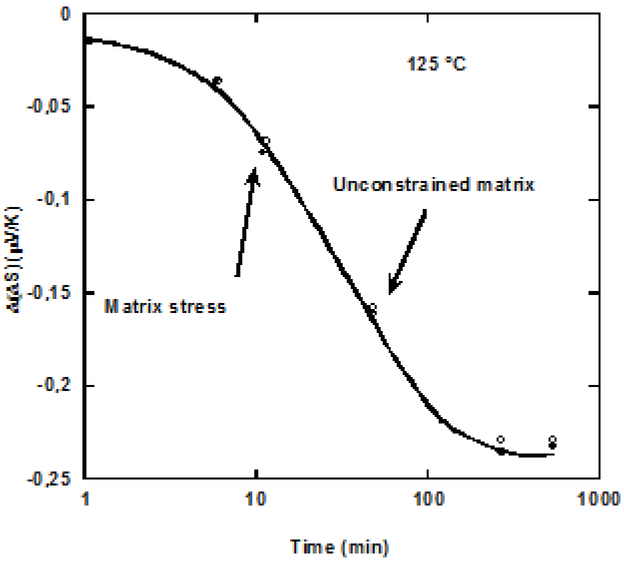

Figure 8: Study of the influence of a uniform stress field on the precipitation kinetics of the β-phase " in 6061. matrix T = 125 ° C

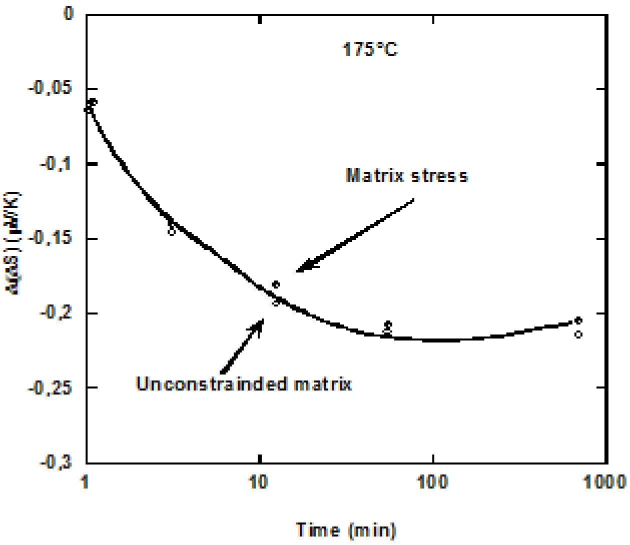

Figure 9: Study of the influence of a uniform stress field on the precipitation kinetics of the β-phase " in 6061. matrix T = 175 ° C.

In addition, we also performed the same comparative experiments using reinforced sample, or not subject to compulsion, for treatment at 175°C. The curves in Figure.10 show no significant difference between the two kinetics. It will be noted in particular that the minimum curve of the PTE is obtained for the same aging time.

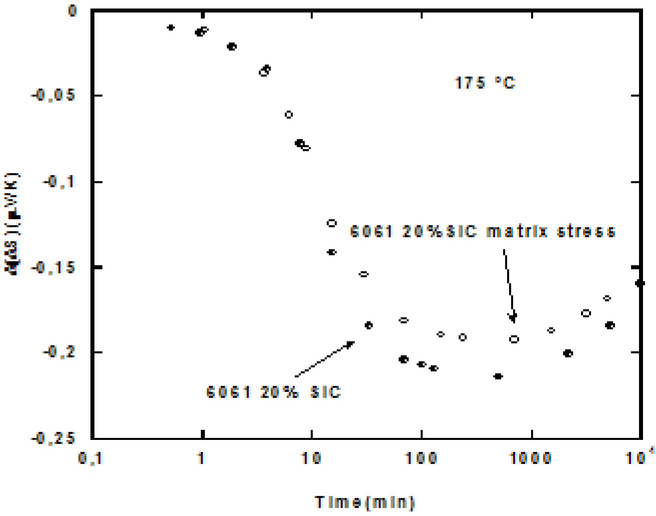

Figure 10: Influence of an external constraint on the precipitation kinetics of the β 'phase' in the composite 6061% / 20 SiCp. T = 175 ° C.

The experiments thus made can not provide indisputable evidence of independence between precipitation and state of internal stress. In particular, we can derive a composite trials gradient constraints have no influence on the precipitation kinetics in the material, since the application of an external stress does not remove these gradients but can not change their distribution or distribution

However, all the results leads to the conclusion logically effect, if any internal stresses on the kinetics of precipitation of second order in the amplitude of the variations.

3.3 Influence of segregations

In the previous literature review (Dafir.1993) , it appears that affected by the segregation of solute around the reinforcement areas, and whose origin was attributed to solute drainage gaps quenching have extensive 'e' to from the rather weak interface (a few hundred nanometers). In a model of spherical particles of radius r and volume fraction f, the volume percentage of the matrix affected by segregation would be: $\phi_v = 3fe / r$

Which, for r = 2.5 microns, 0.2 microns and e = f = 0.2 leads to ϕ_v = 5 %. Given the low volume from the total volume, the influence of these segregations on the kinetics of precipitation does not seem to be very important.

4. Analysis phase precipitation β '.

4.1 Influence of dislocations on the rate of germination

Studies were made on the precipitation phase β ' (Dafir 2012) for aging performed after homogenization followed by quenching to room temperature and studied by the method of cumulative aging time (TVC) , the between successive dipping operations the aging temperature and the room have no effect on the kinetics . In this case, the dislocations may be established during these successive tempers have no significant influence.

However, the kinetics of precipitation is then a function of two parameters:

- The aging temperature which determines the rate of diffusion.
- The quenching temperature which determines the density of dislocation, so the speed of germination of the β -phase.

Thus, the study of the kinetics cannot provide clear information on the microstructural state of the composite during quenching at a given temperature.

The only way to obtain information through the study of the kinetics of precipitation is interrupted to perform quenching temperature aging itself, this temperature being the one for which you want to specify the state of the microstructural composite .

Any time choosing the experimental method (TVC or CDTV) becomes critical. Indeed, we can assume that, in the case of interrupted quenching at high temperature (around 250°C) the rate of dislocation created is quite small, and can increase significantly over the next tempers between this temperature and room temperature. It was therefore necessary to repeat the kinetics obtained by various procedures, time method cumulative aging (TVC) or non-cumulative aging time (CDTV) (SAYED)

Figure.11 compares the sequence of heat treatments carried out in the case of aging after quenching interrupted for two experimental methods (TVC and TVNC). For this type of aging, ΔS_0 reference value of PTE is obtained after initial hardening room of the same sample.

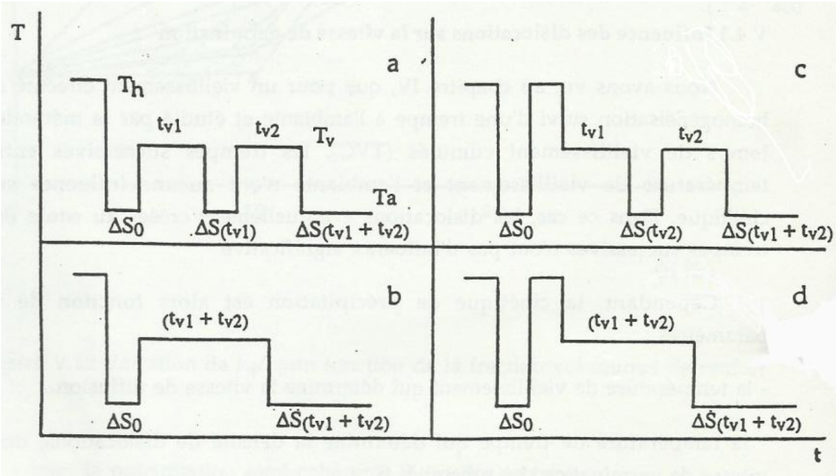

Figure 11: Comparison of heat treatments and TVC TVNC a) and b) water quenching; c) and d) quenching interrupted.

The corresponding results obtained for aging at 250°C, are shown in Figure.12. We observed:

- Speeding up the kinetics of precipitation in the composite relative to that observed in the non-reinforced matrix for both types of treatment
- That the minimum of the curve corresponding to the transition PTE β ' →β is the same for the treatment (TVC) in a water quenching or quenching interrupted (Figure.12.a and b), and this as well as for the composite matrix.
- That, in the case of the composite, the variation curves corresponding PTE to long aging time is the same for both tempers. In contrast, the initial variation of PTE is lower in the case of an interrupted quenching.

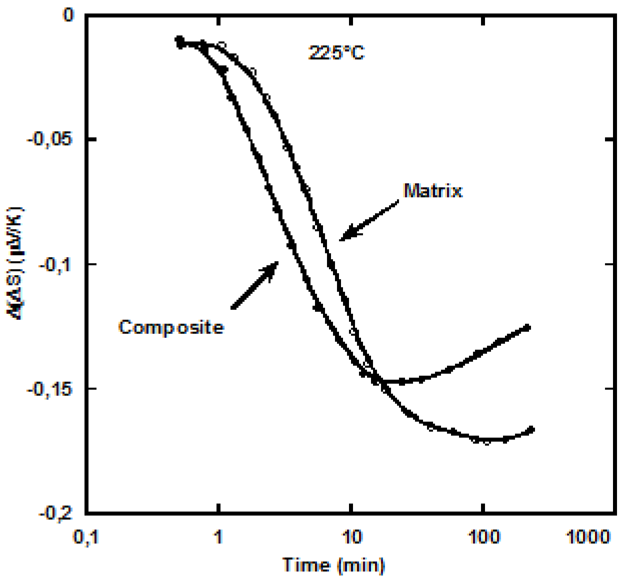

Figure 12 (a): Comparison between TVC TVNC treatment and aging at 250 ° C water quenching

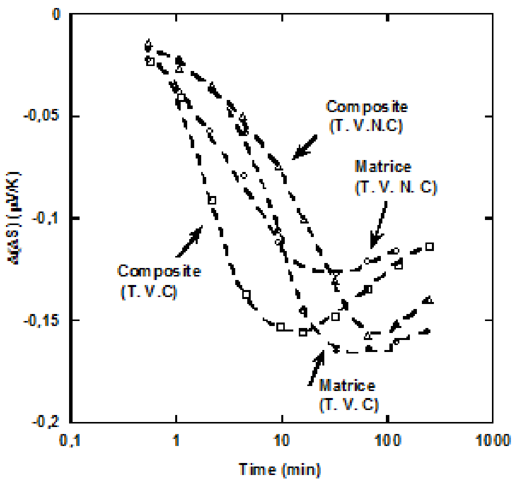

Figure 12 (b): Comparison between TVC TVNC treatment and aging at 250 ° C interrupted quenching

Of these three observations it appears that the speed of germination is heavily modified by the dislocations created at the first hardened at room temperature. This modification is possible because of the initial dislocation density is low, which is not the case when the sample is water quenched.

The kinetics of precipitation aging at a given temperature can therefore, information on the dislocation density introduced into the matrix in the presence of the reinforcement at this temperature, provided that the observations are made on samples aged interrupted after quenching and that the kinetics is followed using the method (CDTV).

4.2 Temperature limit creation of dislocations

We have studied the evolution of PTE in the field of phase precipitation β ' at temperatures income 225°C, 250°C and 275°C (CDTV) method. The Figure.13 shows the variation of PTE according to the aging time at these temperatures, interrupted after quenching the alloy composite and unreinforced. For the three temperatures of aging, there is an acceleration of the precipitation kinetics of the composite compared to the unreinforced matrix..

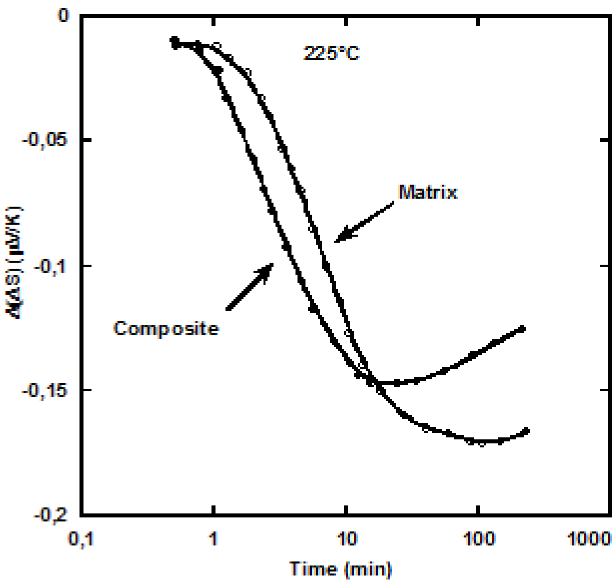

Figure 13 (a): Precipitation Kinetics of β according interrupted quenching. TVNC treatment. Composite 6061/20% SiCp and unreinforced matrix at 225°C.

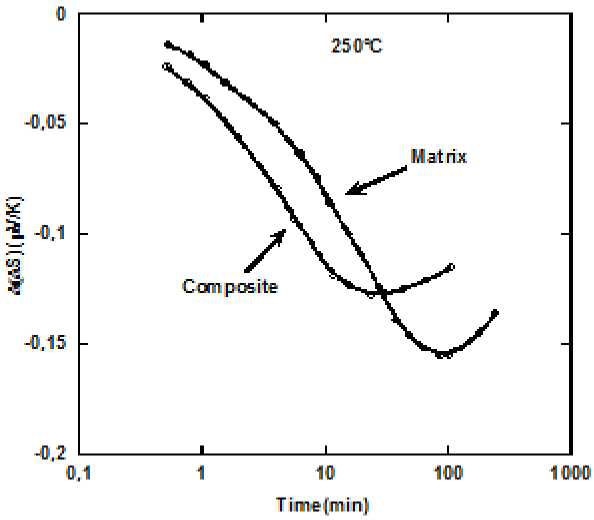

Figure 13 (b): Precipitation Kinetics of β according interrupted Quenching. TVNC treatment. Composite 6061/20% SiCp and unreinforced matrix at 250°C

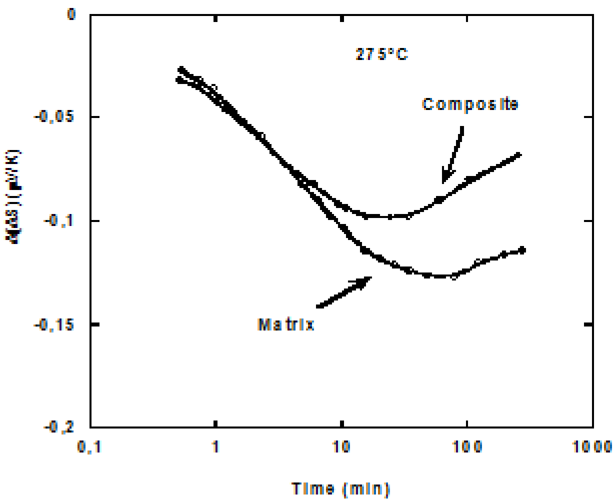

Figure 13 (c): Precipitation Kinetics of β according interrupted quenching. TVNC treatment.Composite 6061/20% SiCp and unreinforced matrix at 275°C.

We then turned to Figure14 transition time t_0 corresponding to the transition β ' β depending 1/Tv. The slope of the line gives, in the case of non-reinforced matrix, an activation of the order of 14 Kcal energy, so that the composite exhibits abnormal behavior athermal. This means that in the latter material, increased diffusion due to the thermal activation, another phenomenon is superimposed having a retarding effect. Since the dislocations have an important influence on the germination of the original semi- coherent phase, we attribute this phenomenon to the timer reduced germination rate due to a decrease of the dislocation density when the aging temperature increases.

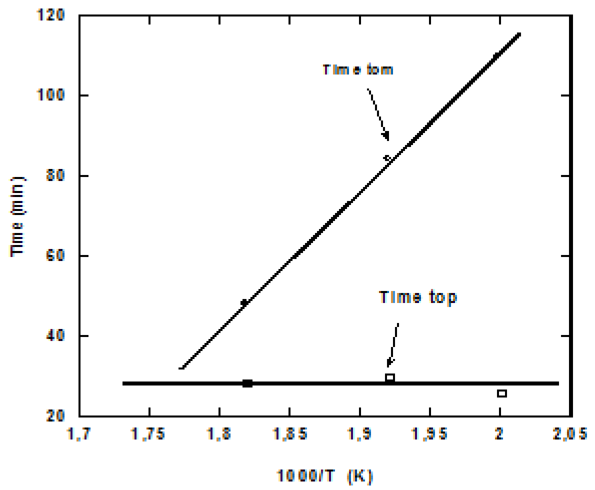

Figure 14: Change in tom / toc and Tv Determination of critical temperature

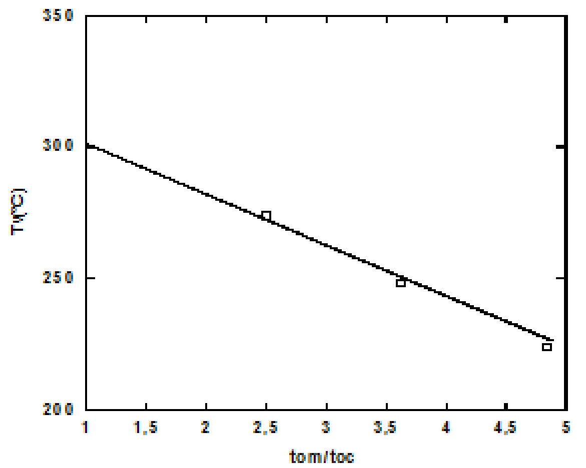

Figure 15: Correlation between tom / toc and Tv Tv Determinationof critical temperature

Figure 15, we have shown the correlation between the annealing temperature and the acceleration of the precipitation in the composite rate, defined by t_{om} / t_{oc}, where tom and toc are respectively the transition time β ' of β and unreinforced matrix composite. This ratio decreases as the aging temperature increases. Extrapolation of the correlation curve to a value of tom report / t_{oc} = 1 allows us to define a critical temperature of about 310 °C, above which there would be no change in the kinetics precipitation in the composite relative to that observed in the non-reinforced matrix. Note that this extrapolation assumes that we neglect in the accelerator effects observed, the contribution of interfaces to that of dislocations. We can therefore assume that above this temperature no dislocation is created by the presence of the reinforcement in the matrix, or, more realistically, is that the dislocations created are restored quickly, either because their density is too low to that we can detect a significant influence on the germination of the phase β'.

This critical temperature value which we found is higher than that obtained by (Vogelsang al.1989) in the case of an alloy 6061renforcé by 20% by volume of SiC particles. Experimental verification of this result is however not possible, on the one hand by the direct phase precipitation β' in the unreinforced matrix cannot be observed at 310°C, secondly by the effects of diffusion interfaces then become dominant.

Given the significance we give to this critical temperature, its value, and not that of the homogenization temperature, which have to serve as a benchmark for evaluating the plastic deformation and residual internal stresses introduced by the presence the reinforcement in the matrix during hardening.

4.3 Analysis of the precipitation of GP zones lacunair

Examination of the kinetics of evolution of PTE between 25 ° C and 100°C (Dafir.19993) can make the following comments:

- For high aging times at 75°C and 100°C , corresponding to the formation of the phase β ', acceleration of the precipitation was observed in the composite according to what has been observed at higher temperatures. Therefore there is no link between the conditions of germination phase β ' (from a pre -precipitation at low temperature (50°C to 100°C) or by direct nucleation from the solution strong in the area 125°C - 200°C, as suggested by the curves of PTE that do not show transitional stage)
- And the accelerating influence of the reinforcement on the precipitation of this phase. This reinforces the hypothesis of an influence of the reinforcement on the growth conditions of the β " and not on the conditions for germination phase.
- Below, this range, the effect of the reinforcement on the precipitation of gaps GP zones is characterized by a reversal in the kinetic behaviour and the unreinforced matrix composite. Indeed there has been an apparent acceleration of the precipitation in the composite at room temperature (see Figure.10) and against , is observed at 75°C (Figure.12), an initial evolution of PTE more low in the composite in the matrix .

The inability to define a late stage (except perhaps at 50°C) and the kinetics normalize however does not allow to refine the case of an actual change in the kinetics and of variation the solute amount in this engaged precipitation phenomenon. This precipitation at low temperature immediately after quenching the sample is fundamentally different from other cases previously discussed, in the sense that the vacancy concentration in the material is not the concentration of thermal equilibrium, but one has instead a very high supersaturation thermal gaps, from the last sample of the high temperature during the homogenization procedure. The kinetics of precipitation observed is therefore highly dependent on the kinetics of elimination of the gaps in supersaturation.

5. Conclusion

Given the complexity of the problem, we can give a qualitative interpretation of the observed phenomena. Micro structural changes due to the presence of the reinforcement in the matrix result in a contradictory double effect:

- An acceleration of the diffusion of the solute due to the modification of the joints or interfaces.

- Inhibition of precipitation due to a more active scavenging gaps super saturation, because of the presence of these interfaces and dislocations created during the quench.

The enthalpy of migration deficiencies in aluminum is about 15 Kcal / mole (1971 Cziraki). The gaps / Mg deficiency or couples / Si are respectively 26 and 33 kcal / mole. An increase in temperature can therefore lead to a significant increase in all of the relative speed of the process of elimination of gaps in relation to precipitation processes such removal shall be effected by a mechanism of diffusion distance. The highly heterogeneous dislocation distribution in the composite (plastic forming a shell around the particles and are not uniformly distributed in the matrix) may be an argument supporting this hypothesis.

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