SPINODAL DECOMPOSITION IN Fe-Cr ALLOYS: EXPERIMENTAL STUDY AT THE ATOMIC LEVEL AND COMPARISON WITH COMPUTER MODELS—III. DEVELOPMENT OF MORPHOLOGY


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Abstract—The fine-scale three-dimensional microstructures formed during spinodal decomposition in Fe-Cr alloys are characterized using two novel methods. In the first, a fractal analysis is used to characterize the interface between the phases and, in the second, the interconnectivity of the structure is determined from topology. It is found that the interface between Fe-rich α and Cr-enriched α′ regions in the experimental data and Monte Carlo simulations exhibit fractal behaviour whereas the microstructures from the solution to the Cahn-Hilliard-Cook model do not. Topological methods are used to characterize the complex α′ microstructures in terms of the number of cavities and loops. The decrease in the number of large scale loops in the microstructure, during thermal ageing, is shown to correlate with the increasing microstructural scale. The number of small scale loops is found to correlate with the complexity of the interface between the α and α′ regions.

1. INTRODUCTION

In this paper, two methods of morphological characterization of percolated structures are considered, fractal and topological analyses. In Part II [1], it was shown that after aging an Fe-45% Cr alloy for 500 h at 773 K, spinodal decomposition had generated structures with a similar scale and composition amplitude to those generated by a Monte Carlo simulation after 5000 MCS and a numerical discretization of the Cahn-Hilliard-Cook equation computed for 1000 time units. Detailed comparisons are made between the microstructures in a series of spinodally decomposed Fe-Cr alloys and computer simulations over these time periods.

2. FRACTAL ANALYSIS

2.1. Introduction

There are several possible methods of characterizing the morphology and topology of complex microstructures. Since fractal geometry can describe highly disordered morphologies, fractal analysis [2-4] might be a suitable tool for analyzing rough interfaces or porous structures. Hornbogen [5] applied fractal analysis techniques to dislocations, grain boundaries, particle distributions and surfaces using optical and electron microscopy. Similar morphologies were found for a wide range of magnifications indicating a fractal characteristic. He also found that many disordered microstructures were not fractal. Unlike mathematical models, a lower and upper cut-off of size scales needed to be introduced, the lower cut-off being determined by the resolution of the imaging technique and the upper by specimen size or grain diameter. Wright and Karlsson [6] considered the applicability of fractal analyses of structures, comparing different measurement procedures on both real surfaces and mathematical models. Their preliminary results indicated that a fractal analysis is useful as an indicator of size, shape and self-similarity but they drew attention to the fact that insufficient statistics limited the accuracy of their results. Cheng et al. [7] used small angle X-ray and neutron scattering experiments to study thin films adsorbed on fractal surfaces by looking at the range of scaling behaviour and deriving a scaling formula for the scattered intensity as a function of film thickness.

Camus [8] suggested that fractal analyses could be used to quantify interconnected microstructures observed by FIM. More recently Miller and Russell [9] have used two-dimensional (2D) digitized field evaporation micrographs to examine the interface morphology of a series of phase separated Fe-Cr specimens. The interface morphology was found to be rough and fractal analysis yielded fractal dimensions...
between 1.1 and 1.5. Hetherington and Miller have discussed the use of fractal analyses in determining an optimum block size at which the composition and other physical parameters may be reliably measured [10].

2.2. Fractal structure

The isosurface representation has already been introduced as a method to represent the interface between two phases (Part I of this series [11]). A series of isosurfaces, generated from the PoSAP analysis of an Fe-45% Cr specimen aged for 500 h at 773 K, is shown in Fig. 1. Each of the surfaces has been generated by a grid with a different resolution. The compositions in each grid cell were calculated using five-point centre-weighted smoothing and the surfaces drawn over every fifth, third, and first points in Fig. 1(a)–(c), respectively. As the resolution becomes finer, more detail is seen and more “particles” appear. This is analogous to the way that the apparent length of a coastline increases with increasing magnification. Choosing the scale on which the isosurface or interface should be defined is equivalent to choosing a coarse-graining volume. From an experimental point of view, it is equivalent to trying to decide which atoms constitute a second phase and which are simply random solute atoms in the matrix phase. Unfortunately, there is no simple solution to this problem, so instead we turn the problem on its head and say that an important characteristic of the structure is that it is not a simple geometric shape. Instead of trying to find an “ideal” length scale, it is accepted that the structure varies with different length scales and then the way in which the structure varies is characterized. The characterization of scaling is the way in which fractals are analysed, and so we can define a “fractal” dimension for the microstructures formed by spinodal decomposition.

2.3. Methods of fractal analysis

It is important to appreciate that there is no single “fractal dimension”. A fractal dimension \( d_f \) may be defined by an equation such as

\[
M(\lambda L) = \lambda^d M(L)
\]

where \( M(L) \) is the mass within a length scale \( L \) and \( \lambda \) is a constant. Using this relation, both diffusion limited aggregation (DLA) and a percolated structure may yield the same value for \( d_f \) (for instance \( d_f = 2.5 \) for an embedding dimension [2] of 3) yet look completely different. There must be some other fractal parameter that differs. Stanley [12] lists ten different measures of dimension necessary to determine some critical exponents for various systems and finds that, as with the critical point exponents, not all the fractal dimensions are independent. Some are connected by simple relations.

A fractal dimension was defined by studying the scaling behaviour of the interface area between the Cr-enriched and Fe-rich regions. A compositional grid was constructed from the data as described in Part I of this series [11] with the use of five-point centre-weighted smoothing. Each grid point was assigned to a particular phase according to the local composition to create a binary array consisting of \( x \) and \( x' \) phases (Fig. 2). The surface area may then be characterized by the number of surface nodes or surface links. In Fig. 2(b), the effect of decreasing the resolution of the image (increasing the size of the ruler over which the grid is defined) is shown. The way in which the surface area of the \( x' \) regions, defined by the number of \( x-x' \) interface links, varies as a function of “ruler length” for an Fe-45% Cr alloy aged for 500 h at 773 K is shown in Fig. 3. The gradient of each plot gives an estimate of one fractal dimension. A normal geometric surface has a dimension of 2 (in an embedding dimension of 3), whereas a completely random structure would have a dimension of 3. To understand this, consider a composition cell belonging to the \( x' \) phase. This cell is considered a surface element if a neighbouring cell belongs to the \( x \) phase. If the volume fraction of the \( x \) phase is \( p_x \) and the structure is random, then the probability that a specific neighbour belongs to the \( x \) phase is simply \( p_x \), irrespective of the length scale chosen. The number of surface cells is therefore proportional to the total number of cells, which is proportional to the volume. Before decomposition occurs, the alloy is quenched from a high temperature above the miscibility gap and therefore has a random structure and each phase has a \( d_f = 3 \) surface. As the alloy ages, well defined domains form and, at the very late stages, the surface dimension must tend towards 2. Therefore, as aging proceeds, the dimension \( d_f \) should decrease from 3 towards 2. The straight line fit (shown in Fig. 3) has a goodness of fit correlation coefficient greater than 0.9995; however the range of “ruler lengths” is limited by the finite size of the analysed volume and the maximum resolution of the microscope. The structures scale over this range, but it has not been proved that they are fractal, since a fractal must be self similar over many decades. It is however an important tool since it characterizes a property of microstructures that is not observed in simple geometric objects such as spheres. Since the fractal dimension varies, it can be used together with other conventional parameters such as wavelength or spinodal amplitude to characterize microstructures.

2.4. Fractal analysis of PoSAP data

Analysis of PoSAP data from the series of Fe-45% Cr alloys thermally aged at 773 K shows a monotonic decrease in fractal dimension of the surface of the Cr-enriched phase with aging [Fig. 4(a)]. Even after 500 h aging, the fractal dimension has only decreased to approx. 2.6, demonstrating that the interface between the domains is not smooth. In order to make an exact comparison between the simulations and experimental results, the effects generated by trajectory aberrations, anode resolution and the finite
Fig. 1 (a and b). (Caption overleaf.)
Fig. 1. PoSAP isosurface reconstructions showing the Cr-enriched regions of an Fe-45% Cr alloy aged for 500 h at 773 K. In (a) every fifth composition point has been used to define the surface. In (b) every third point and in (c) every point has been used.

detection efficiency on the measured fractal dimension need to be ascertained.

2.5. Monte Carlo results

In Fig. 4(b), the decrease in fractal dimension with aging is shown for the Monte Carlo simulation of spinodal decomposition. When the perfect Monte Carlo crystal is analysed, the fractal dimension decreases to a lower value than that observed in the experimental alloys, even though, during the time periods examined, the experimental results and simulations show a similar increase in both scale and composition amplitude (see Part II [1]). However a good match is observed when realistic values of detection efficiency and lateral scatter are used to model the experimental limitations [Fig. 4(b)]. The addition of a Gaussian scatter with a width of 1 atomic spacing to the atomic co-ordinates increases the interface roughness which results in the measurement of a higher fractal dimension.

2.6. Numerical discretization of the Cahn–Hilliard–Cook equation

Fractal analyses of the structures generated by the numerical solution to the Cahn–Hilliard–Cook equation are shown in Fig. 4(c). Three plots are drawn to show the effect of the Cook term, and the finite detection efficiency and lateral resolution of the PoSAP. The Cook term is significant only during the very early stages of aging, before coarsening begins. As decomposition proceeds and the phases begin to coarsen, the fractal dimension rapidly decreases. The fractal dimension measured is close to 2 [Fig. 4(c)]. Even though the scale of the structure is small, the surfaces are ordinary 2D objects in contrast to those observed experimentally or as a result of the Monte Carlo simulation. This results from the fact that the numerical simulation is a continuum model in which the composition is defined on a smooth continuous scale whereas the experimental data and Monte Carlo simulations show the graininess associated with discrete atoms. Even when the effects of lateral scatter,
finite detector resolution and trajectory aberrations are included, the numerical model still predicts much smoother interfaces (i.e. smaller fractal dimension) than are observed experimentally.

3. TOPOLOGICAL ANALYSIS

3.1. Introduction

In Part I of this series [11], isosurface reconstructions were used to show that the phase $\gamma$ often forms a sponge-like interconnected structure. Extending the theories of structure-property relationships to these materials requires a method of characterization in 3D which is equivalent to counting the number of particles for unconnected structures.

In topological analysis, two structures are considered identical if one may be transformed into the other without requiring any cuts or the formation of any closed loops. A coffee cup and ring doughnut are therefore topologically identical. Both structures contain a single handle or loop. Moving a dislocation through a connected microstructure generally requires cutting through the handles of the structure, and therefore the handle density (number of handles or closed loops per unit volume) of a percolated structure is the analogue of the particle density for a system containing isolated particles.

Computing the topological properties of a binary structure is an area of research known as digital topology [13]. Cerezo et al. [14] used topological techniques to characterize 3D interconnected microstructures observed from PoSAP analyses of a range of two phase alloys. They found that a complex morphology could be reduced to a basic framework and characterized in terms of the number of cavities and loops. In this section, the techniques of digital topology have been used to characterize the interconnected structure resulting from spinodal decomposition in the Fe-Cr system and a comparison has been made with the computer models.

3.2. Euler characteristic

Designing a computer program to directly count the handle density in a complex structure would be extremely difficult. However, topology provides us with an indirect method of measuring the handle density, making use of the Euler characteristic $E$.

The Euler characteristic $E(S)$ of a set $S$ is a topological invariant. If $S$ is a polyhedral in 2D then $E(S)$ is equal to the number of connected components of $S$ minus the number of handles in $S$. In 3D $E(S)$ is equal to the number of components of $S$ plus the number of cavities minus the number of tunnels. $E(S)$ may also shown to be equivalent to $(\text{No. of points}) - (\text{No. of edges}) + (\text{No. of triangles}) - (\text{No. of tetrahedra})$. 

![Fig. 2. The area of the interface between two phases can be characterized by the number of surface nodes or links. The area is a function of the grid size used. In (a) every cell is used to define the boundary between the two phases whereas in (b) every other cell is used.](image)

![Fig. 3. Logarithmic-logarithmic plot of surface area measured by the number of surface links against the inverse separation of the points $r$ (in units of grid spacings). The gradient is a measure of fractal dimension. The data are from an Fe-45% Cr alloy aged for 500 h at 773 K.](image)
Several methods of computing the Euler characteristic in 3D have been analysed [15, 16]. Thus in 3D where for instance the number density of handles is related to the interconnectivity of a structure, it is computationally feasible to calculate the number of handles from the Euler relation.

Euler and Poincare [17] proved that for each individual closed surface

\[ E = n - e + f = 2 \]

where \( n \) is the number of nodes on the net, \( f \) the number of faces and \( e \) the number of edges. A simple cube [Fig. 5(a)] has 8 nodes, 12 edges and 6 faces yielding an Euler characteristic of 2. More complicated surfaces may be formed by adding more lines and then applying a topologically invariant transformation. The addition of a line between A and B in Fig. 5(a) creates one new face, three edges and two nodes, leaving the sum \( n - e + f \) unchanged. However, this equation does not hold if the surface encloses tunnels or cavities. If the closed surface \( S \) encloses \( h \) handles and \( c \) cavities (enclosed regions) then

\[ E = n - e + f = 2 - 2h + 2c. \]

For a cube with one handle [Fig. 5(b)], there are 16 nodes, 32 edges and 16 faces, and therefore an Euler characteristic of 0. A cube enclosing a cubic cavity has 16 nodes, 24 edges and 12 faces which yields an Euler characteristic of 4. The latter may be considered as having two closed surfaces (\( E = 2 \) for each) since a cavity is effectively enclosed by a closed surface.

In discussing the skeletonization of 3D digital images, Lobregt et al. used the Euler characteristic to define a connectivity number \( N \)

\[ N = \sum (2 - 2h) \]

summed over all surfaces. The number of closed surfaces minus the number of tunnels is equal to \( N/2 \). A modified version of the algorithm designed by Lobregt et al. [15], which uses lookup tables to store each possible combination of a \( 2 \times 2 \times 2 \) cubic lattice, was implemented. The cavities in the structure are first filled in, which allows the number of surfaces to be calculated simply by counting the number of isolated particles. Connectivity values are then calculated which directly yields the number of handles. Analyses were performed on the simple cubic lattice structures generated by the smoothing algorithms discussed previously in Part II [1].

3.3. PoSAP results

For each material studied, the total number of handles was calculated for the structures generated using both simple and centre-weighted smoothing. The handle density is calculated as the number of handles per atom. The number of atoms field evaporated during the experiment was estimated from the number of ions detected and number of multiple events observed during the analysis but no account was taken for the finite detection efficiency of the channel plates (approx. 60%). In the unaged (quenched) condition, the handle density is very high corresponding to a large number of small handles in the finely percolating random structure. As the material ages, the number of handles decreases (Fig. 6) as diffusion of the atomic species generates a two-phase microstructure whose amplitude and scale are increasing. At lower solute contents, the number of handles is smaller, since the volume fraction of the second phase is smaller. For specimens with a low solute content, isolated particles form with only a few small handles around the interface region.

The results indicate that simple smoothing effectively removes all the very small handles, whereas
the centre-weighted smoothing preserves the fine scale detail associated with the roughness of the interface (Fig. 6). From the difference between the handle densities with the two smoothing techniques, it can be seen that most (approx. 90%) of the handles are very small. There is considerably more scatter on the analysis of alloys with a low solute content. For the series of Fe-17% Cr alloys, the scatter completely masks the decrease in handle density with increasing aging time whereas the trend is clearly shown for the Fe-45% Cr series of thermally aged alloys.

3.4. Monte Carlo simulations

Since the Monte Carlo model simulates atomic diffusion on a perfect lattice it is a relatively simple matter to calculate the number of handles in the microstructure. However, to enable a direct comparison with the experimental results, the finite detector efficiency of the position sensitive detector had to be allowed for. Atoms were randomly removed from the simulations (according to the detection efficiencies for each ion species) before applying the smoothing, as previously described for the analysis of both composition amplitude and measurement of fractal dimensions. The handle density, defined as the number of handles divided by the number of atoms (after removing the “lost” atoms) in the Monte Carlo

Fig. 5. (a) A simple cube (8 nodes, 12 edges and 6 faces). (b) Cube with one handle (16 nodes, 32 edges and 16 faces).

Fig. 6. Handle density for the Fe-45% Cr alloys as a function of aging. Simple smoothing has been applied in (a) and centre-weighted in (b).

Fig. 7. Effect of detector efficiency on measured handle density. Simple smoothing has been applied in (a) and centre-weighted smoothing in (b). The data is from a Monte Carlo simulation performed on a b.c.c. lattice with second nearest neighbour interactions at the equivalent of 750 K. $E_x$ is the detection efficiency for element X.
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Fig. 8. Effect of lateral resolution on handle density. (a) Simple smoothing and (b) centre-weighted smoothing. The simulations were performed on a b.c.c. lattice with second nearest neighbour interactions and the detection efficiencies for Fe and Cr set to 0.5 and 0.3 respectively.

The effect of modelling a decrease in the detector spatial resolution by adding a Gaussian scatter of width \( \sigma \) to the \( x \) and \( y \) position co-ordinates is shown in Fig. 8. In these figures, the detection efficiency of the Fe atoms and Cr atoms was set to 0.5 and 0.3 respectively. After 10 MCS, the scatter is clearly shown to increase the handle density for both simple and centre-weighted smoothing. There are two effects. Mixing in the interface region generates some small scale loops which are calculated with the centre-weighted smoothing. In addition, the number of cells with no composition data increases and the simple smoothing technique fills in the holes and generates links between the phases.

The handle density measured for these structures at intermediate to long aging times is of the same order as that measured for the structures obtained experimentally (Fig. 8). In Table 1, a numerical comparison is made between the experimental results and Monte Carlo simulations on a b.c.c. lattice with second nearest neighbour interactions. A best fit between the Monte Carlo simulations at 750 K and the experimental data occurred when a Gaussian scatter, with width \( s \approx 1 \) atomic spacing, was added to the atomic positions (Table 1).

The curves for the Monte Carlo simulations on a simple cubic lattice at the three temperatures collapse towards a single line when using simple smoothing, suggesting that the overall structures are the same and only the interface structure is different [Fig. 9(a)]. Analysis on the detailed structure (centre-weighted

<table>
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<tr>
<th>PoSAP data Fe-45%Cr</th>
<th>Aging (h)</th>
<th>Handle density (simple smoothing)</th>
<th>PoSAP data Fe-45%Cr</th>
<th>Aging (h)</th>
<th>Handle density (C-W smoothing)</th>
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| Monte Carlo A-50% B on b.c.c. lattice 750 K, \( E_x = 0.5, E_y = 0.3 \) Aging (MCS) | Handle density (simple smoothing) | Monte Carlo A-50% B on b.c.c. lattice 750 K, \( E_x = 0.5, E_y = 0.3 \) Aging (MCS) | Handle density (C-W smoothing) |
|-----------------------------------------------|----------------------------------|----------------------------------|
| \( \sigma = 0 \) | 0.0116 | 0.0112 | 0.0108 |
| \( \sigma = 0.5 \) | 0.0112 | 0.1035 | 0.1025 |
| \( \sigma = 1.0 \) | 0.1025 | 0.1025 | 0.1025 |
| \( \sigma = 2.0 \) | 0.1025 | 0.1025 | 0.1025 |

The curves for the Monte Carlo simulations on a simple cubic lattice at the three temperatures collapse towards a single line when using simple smoothing, suggesting that the overall structures are the same and only the interface structure is different [Fig. 9(a)]. Analysis on the detailed structure (centre-weighted

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The table above provides a comparison between handle density measured from PoSAP data and Monte Carlo simulations. Simple smoothing applied (top) and centre-weighted smoothing applied (bottom).
3.5. Non-linear Cahn-Hilliard-Cook model

The handle density for the structures generated by the solution to the non-linear Cahn-Hilliard-Cook equation is shown in Fig. 10. At early aging times (small amplitude) no order exists and the structure is finely percolated with a high handle density (as is the case for the initial structure in the Monte Carlo simulation—see Fig. 9). An analysis of the autocorrelation function shows that no coarsening occurs before 100 time units. However, during this time period both the number of small scale handles [Fig. 10(b)] and the number of larger handles [Fig. 10(a)] rapidly decreases. Thereafter, the structure coarsens and the number of handles decreases to lower values than observed either from the experimental data or Monte Carlo simulations. This is due to the smoothness of the boundary between the two phases in this model, a direct result of using a continuous composition variable. Even the addition of the Cook term, and the modelling of the detection efficiency and trajectory aberrations, does not generate values as high as those observed from the experimental alloys. A comparison with the Monte Carlo results using four-point simple smoothing, which lacks the fine scale detail of the interface, still shows a different evolution of the handle density. Although the results generated may have a similar scale and amplitude to the Monte Carlo simulations, the details of the interface topology are quite different and so a topological analysis yields different results. The ratio of handle densities decreases with aging, tending towards the limit 1, at which point no small loops exist.

4. DISCUSSION AND CONCLUSIONS

Different measures of fractal dimension can be used to parameterize complex microstructures. The results obtained are sensitive to both detection efficiency and spatial resolution. Data from the experiments and both the simulations showed that the fractal dimension of the interface between the two phases...
temperatures the coarsening process appears to show a small temperature dependence; at higher experimental data [Fig. 12(a)]. The Monte Carlo data (Fig. 12). Again there is some scatter from the structure and handle density after simple smoothing loops.

Digital topology provides a new technique for measuring and characterizing the microstructure in alloys. The Euler relation, and the well developed skeletonization algorithms, linked with the computing power now available, enable a complex morphology to be reduced to a basic framework which may be analysed in terms of the number of cavities and handles. This in turn can be used as a structural parameter for correlation with the mechanical, magnetic or electronic properties of the material. It is therefore important to extend the experimental work to achieve correlation with properties. A high degree of correlation has been found between the experimentally observed Fe-Cr microstructures and those from a Monte Carlo simulation of spinodal decomposition. The smoother interfaces obtained from the numerical solution to the Cahn–Hilliard–Cook equation resulted in the measurement of a lower handle density at later stages of evolution and a decrease in the ratio of handle densities with aging. The information obtained depends on the type of smoothing employed, and, by measuring a handle density, it is possible to characterize the structure in terms of both large scale and small scale loops. The larger loops have been correlated with the scale of the structure and the small loops with interface measurements.

5. CORRELATION OF PARAMETERS

As has been previously stated, the centre-weighted smoothing preserves the fine scale detail, and, with this smoothing, most of the handles measured are small scale loops around the interface region. Since the fractal dimension gives a measure of interface roughness, there should be a relationship between fractal dimension and handle density. To confirm this supposition, a plot of fractal dimension against handle density is shown in Fig. 11. Although the experimental results show some scatter, the trend of decreasing fractal dimension with handle density is clear [Fig. 11(a)]. The Monte Carlo results for a range of temperatures collapse onto a single line [Fig. 11(b)]. The results demonstrate a direct relationship, independent of aging temperature, between the fractal dimension of the interface and the number of fine scale loops.

A similar relationship exists between the scale of a structure and handle density after simple smoothing (Fig. 12). Again there is some scatter from the experimental data [Fig. 12(a)]. The Monte Carlo data shows a small temperature dependence; at higher temperatures the coarsening process appears to proceed slightly more rapidly, whereas the handle density decreases slightly less rapidly. The combination is clearly shown in Fig. 12(b).

It is possible to characterize microstructures in terms of bulk properties (scale of decomposition, number of large scale loops) and interface properties (fractal dimension of the interface area and fine scale loops) as shown in Fig. 12. However, an important question remains unanswered—what principally determines the hardness and embrittlement of these spinodally decomposed alloys? Analysis of both experimental data and the simulations showed that as decomposition proceeds, the scale of the domains increases, the composition amplitude increases and the interfaces between domains become smoother. The relationship between these parameters and the alloy hardness is shown in Fig. 13. Once decomposition had proceeded to such an extent that a scale could be determined, the hardness of the alloys was found to increase linearly with increasing domain size as shown in Fig. 13(a). Figure 13(b) shows the increase in composition amplitude with alloy hardness. Figure 13(c) exhibits a monotonic increase in hardness with
decreasing fractal dimension. Without analysing the effect of heat treatment temperature on the development of scale, composition amplitude and morphology, no firm conclusion can be reached on which parameter gives the best guide to the observed physical properties.

6. CONCLUSIONS

The advances in atom probe microanalysis now enable the 3D reconstruction of composition variations on a sub-nanometre scale. The complex morphology formed during spinodal decomposition of thermally aged Fe–Cr alloys has been analysed. Quantitative parameters have been developed which can fully characterize the domain size, the domain concentrations, the interface morphology and the topology of the microstructure.

Two models of the decomposition process have been implemented and directly compared with the experimental data. Results from the Monte Carlo model accurately matched the experimental data and predicted the observed kinetics of phase separation. The numerical discretization of the Cahn–Hilliard–Cook equation generated qualitatively similar structures, but the domain interfaces were smoother than experimentally observed. Moreover the time exponent for coarsening was found to be higher than the experimental results.

With only a single alloy and single heat treatment temperature it was not possible to determine whether the physical properties exhibited by these alloys are primarily determined by the scale of decomposition, the composition of the phases, the complexity of the interface between the domains or a combination of these parameters. It is therefore important to extend the experimental work to achieve correlation with properties. It will then be possible to use the predictive capability of the models to determine the long term performance and stability of alloys.
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REFERENCES