KAPITEL 4 / *CHAPTER 4* [4]

MECHANISM OF AUSTENITE–CARBIDE COLONY GROWTH IN A DIFFUSION COUPLE

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Introduction

Currently, technological and auxiliary production equipment often operates under increasingly demanding conditions. As a result, metalworking products must exhibit high strength and hardness, as well as possess high plasticity, impact toughness, and excellent wear resistance [1…3]. The development of materials with pre-defined properties has become a prevailing trend in modern materials science. There is a marked interest in designing structurally stable materials that demonstrate an exceptional combination of mechanical properties, including high strength, hardness, and wear resistance, alongside excellent plasticity and impact toughness [4…6]. Composite materials meet the broad spectrum of these elevated requirements and are often described by the conditional mathematical expression: 1+1>2. This expression reflects the synergistic effect, where the combination of the favorable properties of the base components results in a material whose performance exceeds the sum of its parts. This phenomenon has driven growing interest in composite materials [7].

An *in situ* composite is produced in a single-step process from the initial alloy, eliminating the complications associated with combining individual components typical of traditional composite fabrication methods. A classic example of an *in situ* composite is the controlled unidirectional solidification of an eutectic alloy. Eutectics are multiphase composites formed during solidification from the liquid phase [8…11]. The advantage of synthesizing eutectic *in situ* composites during melt crystallization lies in the fact that the resulting structures are not dependent on the properties of the original materials (e.g., particle size or shape), but rather on the solidification conditions. Unidirectional crystallization of an eutectic alloy may lead to the distribution of one phase within another in the form of fibers or lamellae.

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Carburization of iron-based alloys containing carbide-forming, ferrite-stabilizing elements (W, Mo, V, Cr) leads to carbide formation and the recrystallization of ferrite into austenite. It has been shown that carbide formation and recrystallization can occur simultaneously, resulting in the formation of colonial austenite–carbide (γ-C) eutectic-type structures [12…14]. Steels with a colonial structure in the carburized layer exhibit a high level of mechanical properties, wear resistance, and thermal stability. These transformations differ from classical eutectic processes in that they are triggered by a chemical stimulus, not a thermal one.

The publication [15] provides a review of research in the field of studying technologies for obtaining, patterns of structure formation, and properties of natural (*in situ*) composites.

Materials and research methods

The research materials were DIN C110W2 and Fe–20% Cr alloys, whose chemical compositions are presented in Table 1.

Table 1 – Chemical composition of the experimental alloys

Alloy	C, %	Si, %	Mn, %	P, %	S, %	Cr, %	Fe, %
DIN C110W2	1,19	0,21	0,23	0,02	0,01	-	98,34
Fe-20% Cr	0,04	0,07	0,06	0,01	0,02	20,02	79,78

The Fe–20%Cr alloy was produced in an induction furnace using Armco iron as the base material and ferrochrome grade as the chromium source. The melt was cast into steel molds. Cylindrical ingots with a diameter of 85 mm and a length of 330…340 mm were obtained and subsequently used to prepare specimens for further investigation. In the as-cast condition, the microstructure of the alloy consisted predominantly of large ferrite grains with minor amounts of plate-like austenite precipitates located primarily along the grain boundaries. Numerous fine dispersed carbide phase precipitates were observed within the ferrite grains.

Carburization was carried out using a solid-state contact technique. The carbon source was a capsule made of DIN C110W2 steel, into which a conical specimen (1:20 taper) of the Fe–20%Cr alloy was tightly pressed. Structural changes were observed in the alloy in the vicinity of the high-carbon steel capsule. The schematic representation of the diffusion couple is shown in Figure 1. Carburization was performed isothermally at a temperature of 1000 °C. The temperature control accuracy was maintained within ±1 °C. The heating time to reach the target temperature was 30 minutes. The process was conducted in a high-purity argon atmosphere with impurity levels below 0.001%, which effectively prevented the evaporation of alloying elements, particularly chromium, from the sample surface.

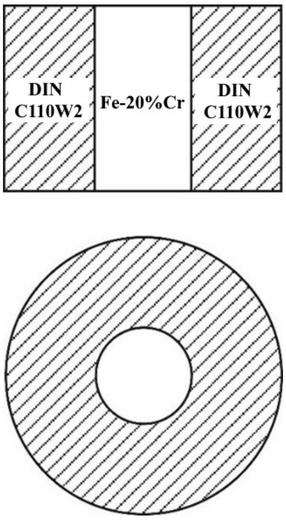

Figure 1 – Schematic of the diffusion couple for studying the growth of austenite–carbide colonies

4.1. Mechanism of nucleation and growth of the carbide phase during carbon diffusion

Carbon diffusion into the specimen from the DIN C110W2 steel capsule begins during the heating stage, prior to reaching the working temperature of the isothermal hold. This process leads to multiple nucleation events and the growth of carbide particles, predominantly of a globular shape, uniformly distributed throughout the matrix volume. In micrographs, the carbide precipitates appear as dark inclusions (Figure 2), which is attributed to the fact that their surfaces are generally not parallel to the polished section and thus scatter light. Simultaneously with carbide precipitation, recrystallization of the ferritic matrix, depleted in chromium and enriched in carbon, into austenite occurs. The growth of austenite–carbide γ-C colonies begins with the appearance of globular carbides along the austenite–ferrite interphase boundary. This initiates the formation of a two-phase growth front that propagates in the direction of carbon diffusion. As a result, columnar γ-C grains are formed, in which the austenitic matrix is penetrated by carbide fibers, primarily plate-like in morphology, with widths corresponding to the transverse dimension of the γ-C grain. A careful examination of sequential micrographs from the same region (Figure 2) reveals that the formation of the γ-C grain structure is localized at this two-phase growth front. Further holding does not lead to qualitative changes in the structure of the formed grains. The transformation front exhibits slight waviness, which is attributed to nonuniform carbon supply. However, the process is self-regulating: in the leading regions of the front, excess carbon promotes the nucleation of new carbide crystals, which consume carbon during their growth. This causes that portion of the front to become a lagging one (Figure 2).

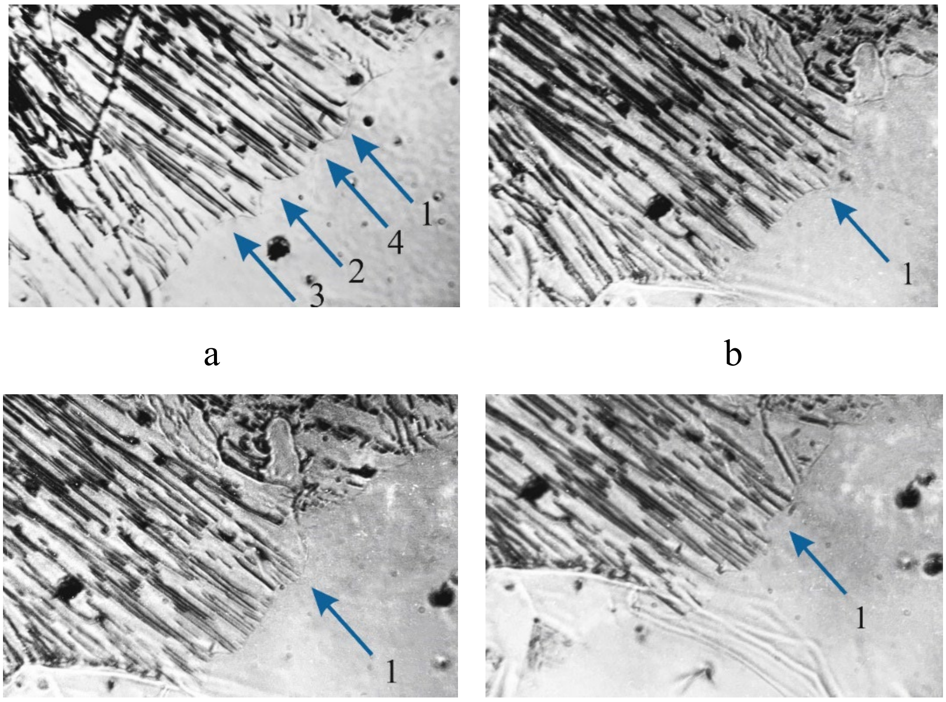

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Figure 2 – Transformation at the growth front of austenite–carbide colonies: (a) τ = 24 min; (b) τ = 31 min; (c) τ = 35 min; (d) τ = 39 min

The interfacial boundary between austenite and ferrite, located between adjacent carbide plates, is typically concave. However, the opposite can also be observed, where carbide fibers slightly advance ahead of the austenitic matrix during growth. This phenomenon is interpreted by some eutectic researchers as evidence that one of the phases (in this case, the carbide phase) acts as the leading phase. Alternatively, this behavior may be explained by the balance of surface tension forces at the triple-phase junction.

According to the findings reported in [16], the transformation front is driven by concentration gradients, which arise from the redistribution of major alloying elements ahead of the recrystallization front.

The main processes occurring at the growth front of the γ-C colonies that determine their structural evolution are the nucleation of new carbide fibers and the extinction of old ones. Typically, new fibers emerge in the spaces between diverging plates (arrows 2 and 3 in Figure 2), while fibers tend to extinction in the convergence zones of adjacent plates (arrow 4 in Figure 2). This dynamic establishes a characteristic average inter-plate spacing. During γ-C colony growth, both fiber nucleation and extinction occur continuously, meaning that any correlation between the front propagation rate and the inter-plate distance can only be made in terms of an average inter-plate spacing which tends to increase as colony grows and the front propagation rate decreases.

Ferrite grain boundaries are crossed by growing colonies without significant structural disturbances or changes in growth rate. However, many growing fibers die off, so the inter-plate spacing distance increases discontinuously. The situation changes if the passage of γ-C colonies across the boundary is preceded by austenite precipitation. In such cases, γ-C grains cease growing upon reaching the austenitic boundary layer (Figure 3). Then growth resumes, but the morphology of the γ-C grains may undergo significant alteration. For example, rod-like colonies may form instead of plate-like ones, indicating that the passage through the austenite interlayer induces the nucleation and growth of new γ-C grains. The resulting grain morphology is largely governed by the crystallographic orientation of the austenite relative to the growth direction of the γ-C colonies, and thus by the interfacial energy parameters between austenite and carbide phases. Upon passing through subsequent austenite interlayers, a qualitative change in grain morphology may be observed again, for instance, rod-like colonies may transform back into plate-like ones (Figure 3).

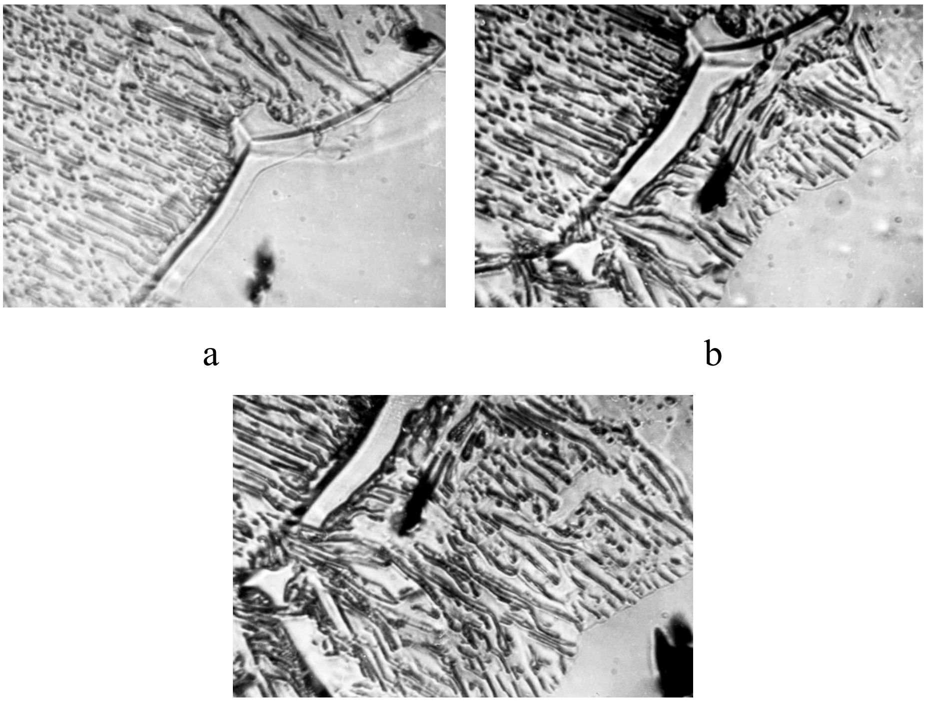

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Figure 3 – Penetration of ferrite grain boundary with austenite interlayer by austenite–carbide colonies: (a) τ = 78 min; (b) τ = 99 min; (c) τ = 110 min

4.2. Influence of temperature variations on the growth of γ-C colonies during contact carburization of an iron–chromium alloy

The iron–chromium alloy system is particularly advantageous for investigation as the formation of γ-C colonies occurs within a wide temperature range, especially when compared to iron-based alloys with molybdenum, vanadium, tungsten, or titanium. In the investigated alloy containing 20% Cr, γ-C colony formation occurs within a temperature range of 850–1000 °C. A base temperature of 900 °C was selected for the experiment, at which stable growth of γ-C colonies with a relatively smooth and uniform transformation front was observed (Figure 4 a). Then the temperature was gradually decreased, first to 890 °C, and then, five minutes later, to 880 °C. A modest decrease of only 10 °C triggered the nucleation of numerous new carbide particles at the transformation front, predominantly in areas of initial coarse differentiation. As a result, the differentiation of γ-C colonies becomes more refined (Figure 4 b).

A further decrease by another 10 °C led to additional refinement of differentiation. At this stage, the nucleation of new carbide particles at the two-phase transformation front was observed to occur through branching or splitting of existing carbide fibers that grow (Figure 4 c). Increasing the temperature back to the base level of 900 °C results in the appearance of an austenite interlayer (Figure 4 d).

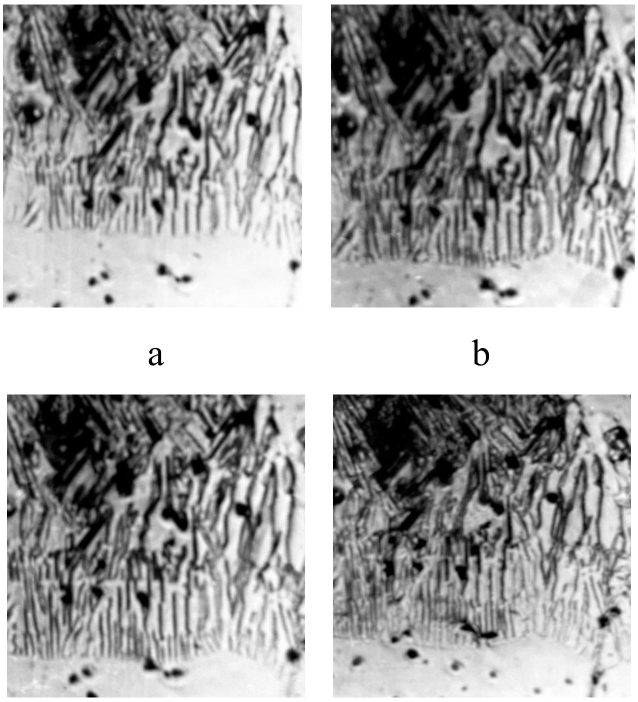

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Figure 4 – Transformation at the growth front of austenite–carbide colonies during temperature decrease in the carburization process: (a) *t* = 900–890 °C, τ = 78 min; (b) *t* = 890 °C, τ = 81.5 min; (c) *t* = 880 °C, τ = 86.5 min; (d) *t* = 900 °C, τ = 107 min.

Upon reaching the working temperature for isothermal holding, and before cooperative transformation into γ-C colonies begins, initial preferential carbide precipitation gives way to recrystallization, resulting in the formation of a single-phase austenitic zone. Only then do the two-phase γ-C columnar structures occur. This sequential structural evolution is characteristic of the surface region of the sample during carburization. However, when the transformation occurs within the bulk, examination of longitudinal sections of the carburized layer reveals that columnar structures directly follow regions with dispersed carbide precipitates, without any intervening austenite layer. This behavior can be explained by the accelerated diffusion of the impurity (in this case, chromium) along the surface of the polished section toward the areas of the solid solution that have been depleted due to extensive carbide precipitation. Carburization of chromium-depleted alloy, especially at elevated temperatures, may lead to direct recrystallization of ferrite into austenite without carbide formation. It is more plausible that the formation of an austenite interlayer results from rapid heating of previously carburized chromium-containing ferrite at lower temperatures.

4.3. Mechanism of γ-C colony formation

Figure 5 demonstrates that the carbide phase evolves through dendritic branching from a central core. Initially, the growth direction of carbide fibers is not aligned with the carbon diffusion flux (indicated by the arrow), but as growth proceeds, the fibers become elongated in the direction of carbon diffusion, resulting in coarser colony differentiation. In certain regions of the polished section (Figure 6), one can clearly observe the splitting of carbide fibers due to temperature decrease after isothermal holding, which leads to refinement of colony structure (arrow 1) and the coarsening of colony differentiation upon subsequent temperature increase, as many fibers cease growing (arrow 2).

The presented direct observations of the growth of γ-C colonies make it possible to explain specific features of columnar structure formation, in particular, the formation of austenitic interlayers due to temperature variations (Figure 6) and change in differentiation.

An increase in the amplitude of thermal cycling during cooling initially causes a refinement of the differentiation within the γ-C colonies, followed by carbide growth along the growth front and eventual cessation of its advancement. Upon reheating, the

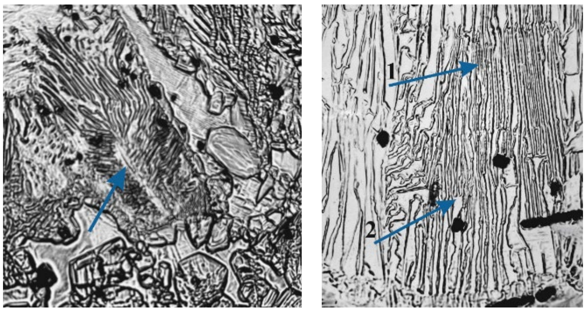

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Figure 5 – Microstructure of the carburized layer of the sample after high-temperature metallography experiments: (a) ×400, (b) ×630

Figure 6 – Structural changes at the growth front of austenite-carbide colonies during thermal cycling; electrolytic etching; ×630

formation of an austenitic layer precedes the resumption of γ-C colony growth. At the early stages, carbide growth is irregular, but regular colony growth reestablishes with time.

When the interlamellar (or interrod) spacing exceeds that which corresponds to the maximum growth rate, the affected regions lag behind and experience a relatively enhanced carbon supply compared to the more advanced front segments that move quicker. This lagging occurs because at larger spacings, the necessary redistribution of alloying elements (chromium, in this case) cannot keep pace. Mother phase (ferrite) ahead of the austenite is enriched in chromium, while regions ahead of the carbides are depleted. Carbon concentration required for transformation in ferrite increases near the transformation front due to the inhibited redistribution of chromium, which stabilizes the mother phase. Given that chromium diffuses much more slowly than carbon and that chromium enrichment/depletion in ferrite is confined to a narrow zone, thermodynamic instability of ferrite increases with distance from the transformation front. This phenomenon resembles concentration undercooling, whereby ferrite ahead of the two-phase front becomes more supersaturated with carbon than at the front itself. Directly at the transformation front, enrichment of alloying elements ahead of the austenitic phase promotes carbide formation, whereas depletion ahead of the carbide phase favors ferrite recrystallization into austenite.

Under such conditions, new carbide fibers nucleate and grow, resulting in decreased interlamellar spacing. According to direct observations, new fibers emerge by branching of existing fibers and by nucleation of new carbide crystals. This effect is pronounced when carbon supersaturation of ferrite is increased by lowering the carburizing temperature.

Conversely, if the growth rate is lower than the maximum possible due to small interlamellar spacing, the growth rate reduction is caused by capillary forces at the carbide-austenite interface. These forces raise the equilibrium carbon concentration in ferrite so the transformation front can stop. In this case, interaction with neighboring faster-advancing regions can cause some carbide fibers to cease growth randomly. Consequently, the carbide-ferrite portion of the front transforms into an austenite-ferrite region, increasing the interlamellar spacing and thereby accelerating the growth rate. The process becomes clearly evident when the decrease in carbon supersaturation of ferrite ahead of the transformation front is caused by an increase in temperature during carburization.

The combined theoretical and experimental evidence indicates that the formation of colonial austenite-carbide structures proceeds through a simultaneous and joint transformation of carbon-supersaturated ferrite into austenite and carbide precipitation. This is an isothermal transformation requiring external carbon supply. Morphologically and mechanistically, it resembles eutectic or eutectoid transformation, with component redistribution occurring via diffusion along the moving two-phase interface between neighboring growing phases.

Summary and conclusions.

Carburizing of iron-based alloys containing carbide-forming elements induces carbide precipitation and ferrite recrystallization into austenite.

The ferrite decomposition into austenite and carbide, driven by carburizing, represents a phase transformation that can be exploited to produce *in situ* composites with carbide reinforcement in iron alloys.

Using the diffusion couple of DIN C110W2 and Fe-20%Cr alloys, simultaneous carbide precipitation and ferrite recrystallization into austenite during compositional diffusion has been demonstrated. This leads to the formation of colonial austenite-carbide structures, which constitute natural composites.

Interlamellar spacing can increase by coalescence of growing carbide fibers or by detachment and cessation of growth of individual fibers. Conversely, spacing decreases via fiber branching and nucleation of new carbide fibers at the austenite-ferrite interface boundary.

Strict control of isothermal conditions is essential to stabilize the transformation process, as minor temperature variations cause disturbances at the transformation front leading to austenitic layer formation during heating and carbide layer formation during cooling, thereby disrupting the continuity and regularity of the austenite-carbide grains.