Microstructure Evolution during the Solidification of Steel

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In today's material world it is agreed that a complete understanding of the solidification path and of the resulting microstructure can only be obtained by treating the alloys as mathematical systems, followed by validation of the models through definitive experiments. This paper will attempt to review the main recent experimental and mathematical efforts directed to the understanding of microstructure evolution in steel.

KEY WORDS: microstructure evolution; steel; peritectics; solidification; modeling.

1. Introduction

Iron-base alloys, that include steel and cast iron, are some of the oldest man made materials. However, they have enjoyed a remarkable longevity because of their superior mechanical and physical properties, coupled with their competitive price. They are the most widely used casting materials. Since properties are directly related to microstructure, a significant amount of research has been directed to the understanding of the evolution of steel microstructure during solidification and subsequent cooling to room temperature. While steels are in principle solid solution alloys, their microstructure evolution during solidification is complicated by the existence of a peritectic. Many other metallic, ceramic and organic materials exhibit a peritectic transition including Fe–Ni, bronze (Cu–Sn), brass (Cu–Zn), Al–Ti, lanthanide magnets (Nd–Fe–B) and ceramic superconductors (Y–Ba–Cu–O). Some of these materials have exotic applications. For example, the naphthalene-capric acid system is a potential latent heat storage material. As shown in Fig. 1, at temperatures less than 1498°C the solidification microstructure of low-carbon steel is single phase austenite. Yet, depending on the carbon content the solidification path can be quite different. At carbon contents less than 0.16%, at the end of peritectic solidification both δ ferrite and γ phase coexist, while over 0.16% C liquid and γ coexist. At carbon contents higher than 0.53% only austenite solidifies from the liquid. Also shown on the figure are the metastable extensions of the γ phase that could form at any composition directly from the liquid if nucleation of the δ phase is suppressed. Such suppression of the properitectic phase has been demonstrated in a number of peritectic binary melts, including Fe–Mo, Co–Si and Al–Co alloys.

2. Mechanism of Peritectic Solidification

The peritectic solidification starts with a 'peritectic reaction' in which all three phases, δ, γ, and liquid are in contact with each other (Fig. 2). The peritectic γ phase will grow along the solid/liquid (S/L) interface δ/L, driven by liquid super-saturation. Solute rejected by the γ phase will diffuse through the liquid to the δ phase contributing to its dissolution. The γ phase will also thicken in the direction perpendicular to its growth, by direct growth in the liquid and at the expense of the δ phase by solid state diffusion. Once the reaction is completed and all the δ/L interface is covered by γ, the 'peritectic transformation' starts. The liquid and the primary δ phase are isolated by the γ phase. The transformation δ→γ takes place by long-range solid-state diffusion through the peritectic γ phase. The γ phase grows by direct solidification in the liquid. According to Fredriksson, under certain conditions it is possible for nucleation and growth of the γ crystals to occur in the liquid without contact with the δ crystals. Following nucleation, the secondary phase grows freely in the liquid, while the primary phase dissolves.

3. Peritectic Microstructures

Direct evidence of these mechanisms has been provided recently through in situ dynamic observation of the
progress of peritectic reactions and transformations of Fe–C alloys made with a combination of a confocal scanning laser microscope and an infrared image furnace. Selected micrographs are presented in Fig. 3. It is observed that as the thermal gradient decreases from 22 K/mm (left column) to 4.3 K/mm (middle column) the δ solid/liquid interface becomes unstable and changes from planar to cellular. Further increase in solidification velocity from 2.5 to 19.3 μm/s showed that γ phase starts growing at the boundaries of the δ cells. Upon further increase of velocity to 38.7 mm/s, island-like δ crystals appeared (Fig. 3, right column, a) which then underwent peritectic reaction and transformation (Fig. 3, right column, c). The wrinkles observed on the γ crystals that transformed from the δ crystals are thought to be due to the volume contraction of the transformation.

Similar observations were also made for the Fe–Ni system. The two stages of the peritectic transition involving the reaction (austenite growing along the liquid–ferrite interface) and the transformation (direct solidification of austenite from the liquid) were observed.

In general, a variety of microstructures can result from peritectic solidification, mostly depending on the temperature gradient/solidification velocity ($G_T/V$) ratio and nucleation conditions. The possible structures include cellular, plane-front, bands, eutectic-like structures.

Simultaneous growth of two phases as oriented fibers and lamellae has been observed in some peritectic alloys when the composition was on the tie-line of the two solid phases and the $G_T/V$ ratio was close to the limit of constitutional undercooling for the stable phase having the smaller distribution coefficient. An example of such a structure for a Fe–Ni alloy is presented in Fig. 4.

Banded structures have been observed in peritectic alloys at low growth rates. An example is provided in Fig. 5(a). The formation of bands is explained by nucleation and growth of the second phase during the initial transient of planar growth of the primary phase and vice versa. This occurs because the liquid at and ahead of the growing inter-
face is constitutionally undercooled with respect to the other phase. As the second phase nucleates and grows ahead of the primary phase, the former phase cannot reach the steady state. Similarly, the primary phase nucleates again during the transient growth regime of the second phase, preventing it for reaching the steady state. Consequently, a cycle is set up leading to the layered microstructure.\(^{10,11}\)

Several other structures can be obtained depending on the relative importance of nucleation diffusion and convection (Fig. 5).\(^ {12}\) Theoretical models and experimental studies in thin samples suggest that the structures (a)–(e) can form under diffusive regime, while microstructure (f) requires the presence of oscillatory convection in the melt.

In an attempt to rationalize this plethora of peritectic microstructures, prediction of phase and microstructure selection was attempted by generating microstructure selection diagrams for peritectics. The main variables controlling microstructure evolution include interface velocity \(V\), thermal gradient \(G\), alloy composition \(C\) and nucleation potential.

Assuming that the leading phase, that is the phase that grows at the highest interface temperature is the kinetically most stable one, Umeda \(^{1}\) et al. developed an equation that describes the transition velocity from \(\delta\) dendrites to \(\gamma\) dendrites in directionally solidified alloys:

\[
V_{\delta\rightarrow\gamma}^{\alpha} = \frac{D_L}{4\pi} \left( \frac{\Delta T_{m}^{\delta\rightarrow\gamma} - C_{\alpha} \Delta m_{\delta\rightarrow\gamma}^{\alpha}}{(C_{\alpha} m_{\delta}^{\alpha})^{1/2} - (C_{\alpha} m_{\gamma}^{\alpha})^{1/2}} \right)
\]

(1)

where \(D_L\) is the liquid diffusivity, \(\Delta T_{m}^{\delta\rightarrow\gamma}\) and \(\Delta m_{\delta\rightarrow\gamma}^{\alpha}\) are the melting point difference and the liquidus slope difference between \(\delta\) and \(\gamma\), respectively, and \(k_\delta\) and \(\Gamma_\gamma\) are the partition coefficient and the Gibbs–Thomson coefficient of the \(\gamma\) or \(\delta\) phase. The numerator in the parenthesis represents the difference in growth kinetics between the dendrites of the two phases. This equation was plotted in Fig. 6(b) as a function of composition for Fe–Ni alloys whose phase diagram is presented in Fig. 6(a). It is noticed that for a given composition of 4.2 at\% Ni, as the interface velocity increases, a transition from \(\delta\) to \(\gamma\) dendrites occurs at about \(8 \times 10^{-2}\) m/s.

The approach described in the previous paragraphs cannot explain band formation, which is apparently the result of nucleation and growth of the second phase during the initial transient of planar solidification of the primary phase and vice-versa. According to Trivedi,\(^ {10}\) the liquid ahead of the growing interface is constitutionally undercooled with respect to the other phase. As the second phase nucleates and grows ahead of the primary phase, the former phase cannot reach steady state. Then, the primary phase nucleates ahead of the growing second phase preventing it from reaching steady state. Thus, a cycle leading to banded microstructure is set up.

By combining the maximum growth temperature criterion with nucleation considerations, Hunziker \(^{13}\) et al. developed a microstructure selection diagram for peritectic alloys close to the limit of constitutional undercooling. The diagram, presented in Fig. 6(c) for Fe–Ni alloys, assumes negligible nucleation undercooling for both \(\delta\) and \(\gamma\) phases and allows prediction of planar front, cellular, dendritic, and band solidification. The transition lines on the \(G/V\)–\%Ni graph are calculated with the equations presented in the following text and plotted on Fig. 6(c).

The transition from planar-to-cellular growth of the \(\delta\) phase is given by the limit of constitutional undercooling:

\[
\frac{G}{V} \approx -\frac{m_\delta^2(C_{\alpha} - C_{\delta}) - m_\gamma^2(C_{\gamma} - C_{\delta})}{D_L} \quad ...........(2)
\]

where \(C_{\delta}\) is the composition of the liquid at the equilibrium peritectic temperature and \(m_\delta^2\) is the solidus slope of the \(\delta\) phase. By substituting the superscript \(\delta\) with \(\gamma\), the equa-
tion can be adapted to describe the transition from planar-to-cellular growth of the \( \gamma \) phase.

The stability condition for planar \( \delta \) with respect to \( \gamma \) nucleation was calculated by comparing the interface temperature with the nucleation temperature of \( \gamma \), which resulted in the following equation:

\[
C_o < C_\delta - \frac{m_\delta \Delta T_N^\gamma}{m_\delta^2 (m_\delta^2 - m_\gamma^2)} \tag{3}
\]

where \( \Delta T_N^\gamma \) is the nucleation undercooling. Note that when the nucleation undercooling is negligible the condition reduces to \( C_o < C_\delta \), as shown in Fig. 6(c).

Similarly, by comparing the interface temperature with the nucleation temperature of \( \gamma \), the stability condition for cellular \( \delta \) with respect to \( \gamma \) nucleation was derived as:

\[
\frac{G}{V} < \frac{m_\delta^2}{D_L} (C_o - C_\gamma) + \frac{\Delta T_N^\gamma}{m_\delta^2 - m_\gamma^2} \tag{4}
\]

The stability condition for planar \( \gamma \) with respect to \( \delta \) is given by and equation similar to Eq. (3):

\[
C_o > C_\gamma - \frac{m_\gamma \Delta T_N^\delta}{m_\gamma^2 (m_\gamma^2 - m_\delta^2)} \tag{5}
\]

When the nucleation undercooling is negligible this stability condition reduces to \( C_o > C_\gamma \), as shown in Fig. 6(c).

Additional stability conditions that will not be presented here are invoked to derive the stability condition between the two phases in the cellular regime:

\[
\frac{G}{V} > \frac{(m_\delta^2 - m_\gamma^2)(C_o - C_\gamma) - \Delta T_N^\gamma}{D_L \ln(m_\gamma^2 / m_\delta^2)} \tag{6}
\]

In the region marked “bands” on Fig. 6, neither \( \delta \) nor \( \gamma \) are stable at steady state, and either phase can nucleate ahead of the other’s plane front. This is the condition for bands formation. In the region marked “mixed bands” alternate layers of cellular \( \delta \) and planar \( \gamma \) are expected to form.

Nucleation undercooling can significantly affect the extent of the bands regions. Indeed, as the nucleation undercooling increases the stability lines defined by Eq. (3) and Eq. (4) move to the right, while that defined by Eq. (5) moves to the left.

Finally, a comparison between predictions with the maximum growth temperature criterion and the combined maximum growth temperature–nucleation model are summarized in Fig. 7. The importance of nucleation becomes clearly evident.

4. Analytical Modeling of Peritectic Solidification

A number of analytical models have been proposed. Their goal is to describe quantitatively the peritectic reaction, the peritectic transformation and some of the more unusual structures such as the banded structure.

4.1. The Rate of the Peritectic Reaction

The peritectic reaction, which is the propagation of the triple point \( L/\gamma/\delta \) along the \( L/\delta \) boundary of planar \( \delta \) crystals, consists of the dissolution of the \( \delta \) phase and growth of the \( \gamma \) phase. It is controlled by the growth of \( \gamma \) since dissolution is the fastest process. Bosze and Trivedi\textsuperscript{14} simplified an earlier equation developed by Trivedi to describe the relative contributions of diffusion, surface energy and interface kinetics during the growth of parabolic shape precipitates. Using their model it appears that the peritectic reaction is controlled by undercooling and liquid diffusivity according to the equations:

\[
\frac{G}{V} < \frac{m_\delta^2}{D_L} (C_o - C_\gamma) + \frac{\Delta T_N^\gamma}{m_\delta^2 - m_\gamma^2}
\]

\[
\frac{G}{V} > \frac{(m_\delta^2 - m_\gamma^2)(C_o - C_\gamma) - \Delta T_N^\gamma}{D_L \ln(m_\gamma^2 / m_\delta^2)}
\]
with 

where \( r \) is the radius of the leading edge (the plate will have a thickness of \( 2r \)), and \( C_{ij} \) are interface concentrations (see Fig. 8 for definitions).

Using the maximum growth rate theory \(^{14,15}\) it can be shown that the thickness of \( g \) increases with lower solidification velocity and with larger surface energy difference (see Fig. 9):

\[
\frac{D_g}{H_1} = \frac{g_L}{H_2} \frac{g_d}{H_2} \]

\[
\Omega = \frac{C_{Ly} - C_{Ld}}{C_{Lg} - C_{Lg}} \quad \text{...........................(8)}
\]

where \( r \) is the radius of the leading edge (the plate will have a thickness of \( 2r \)), and \( C_{ij} \) are interface concentrations (see Fig. 8 for definitions).

Using the maximum growth rate theory \(^{14,15}\) it can be shown that the thickness of \( \gamma \) increases with lower solidification velocity and with larger surface energy difference (see Fig. 9):

\[
\Delta \gamma = \gamma^L \gamma^L + \gamma^\delta \gamma^\delta \quad \text{...........................(8)}
\]

However, \textit{in situ} observation in Fe–C systems \(^6\) showed that the experimental velocities were much higher than the ones predicted with this model. This suggests that the peritectic reaction is not controlled by carbon diffusivity in the liquid, but perhaps by either massive transformation of \( \delta \) into \( \gamma \), or direct solidification of \( \gamma \) from the liquid. Support of these hypotheses was brought recently by the experimental work of Dhindaw \textit{et al.}\(^{16}\) who studied the peritectic reaction in medium-alloy steel (0.22 mass% C, 1.3 mass% Cr, 2.6 mass% Ni). Microsegregation measurements on directionally and isothermally solidified samples showed that when the segregation ratio for Ni was higher than that for Cr a peritectic reaction has occurred. However, when the segregation ratio for Cr was higher than for Ni, the liquid was transformed directly into \( \gamma \) without undergoing a peritectic reaction. Based on the evaluation of the energy of transformation through differential thermal analysis the authors concluded that the transformation is a diffusionless transformation \( \delta \rightarrow \gamma \).

4.2. The Rate of the Peritectic Transformation

Assuming isothermal transformation and growth of \( \gamma \) layer controlled by carbon diffusion, Hillert\(^{17}\) proposed that the thickness of the \( \gamma \) layer can be calculated as:

\[
\Delta x_\gamma = [2D_\gamma (\Omega_{\gamma L} + \Omega_{\gamma L}) t]^{1/2} \quad \text{with}
\]

\[
\Omega_{\gamma L} = \frac{C_{gL} - C_{gL}}{C_{gL} - C_{gL}} \quad \text{and} \quad \Omega_{\gamma L} = \frac{C_{gL} - C_{gL}}{C_{gL} - C_{gL}} \quad \text{...........................(9)}
\]

where \( D_\gamma \) is the diffusivity in the \( \gamma \) phase, \( t \) is time and \( C_{ij} \) are interface concentrations (see Fig. 10 for definitions). This equation suggests that the thickness of the \( \gamma \) phase and the growth rate increase with higher undercooling and diffusivity.

For substitutionally dissolved elements (e.g., Fe–Ni) the growth rate will be very low. Under normal casting conditions the amount of \( \gamma \) phase formed through peritectic transformation will be negligible in comparison with that formed through precipitation from the liquid. However, for the Fe–Ni system it was found that the solidification rate was a function of the local temperature gradient rather than undercooling.\(^7\)

For interstitially dissolved elements (e.g., Fe–C) the diffusivity rates are much higher and the peritectic transformation is completed within 6 to 10 K of the equilibrium temperature.\(^{15,18}\) The rate controlling phenomenon is carbon diffusion. Indeed, \textit{in situ} observation in Fe–C systems \(^6\) demonstrated that the growth of the thickness of the \( \gamma \) phase follows a parabolic law which supports the opinion that carbon diffusion determines growth rate. Calculations with a simple finite difference model\(^{19}\) showed good agreement between calculated and experimental migration distances of the \( \gamma / \delta \) and \( \gamma / L \) interfaces in time.

Fredriksson and Stjerndahl\(^{20}\) expanded Hillert’s model to continuous cooling by assuming that the boundary conditions change during cooling and that the cooling rate is constant. From the isothermal equation they calculated that the thickness of the \( \gamma \) layer is given by:
\[ \Delta x = c_t \cdot \left( \frac{dT}{dh} \right)^{-1} \cdot \Delta T_p \] ..................................(10)

This equation was used to plot the peritectic temperature range as a function of the carbon content (see Fig. 11). It is seen that the reaction is relatively fast and is finished at maximum 6 or 10 K below the peritectic temperature, depending on the cooling rate.

4.3. Growth of Banded (Layered) Peritectic Structure

The formation of layered structures has been observed in several peritectic systems including Sn–Cd, Sn–Sb, Zn–Cu, Ag–Zn and Pb–Bi. Layered structures have been observed in both hypo-peritectic and hyper-peritectic systems, but always at high G/V ratios. In principle the banded structure results when the second phase nucleates ahead of the planar primary phase. If the lateral growth of the secondary phase is higher than the normal growth of the primary phase, a planar band of secondary phase will form ahead of the planar primary phase. If lateral growth is slower than normal growth, incomplete bands will result. Some typical microstructures for layered growth are given in Figs. 5(a), 5(b), and 5(f) and in Fig. 12.

To determine conditions that control the volume fraction and spacing of bands Trivedi has developed a model based on the following assumptions: solute transport by liquid diffusion only; no liquid convection; negligible diffusion in solid; growth conditions are such as to produce planar S/L interface. The widths of the \( \alpha \) and \( \beta \) layers were derived to be:

\[ \lambda_i = \frac{D_i}{V} \ln A_i \] ..................................(11)

where \( i = \alpha \) or \( \beta \), the concentrations and slopes are defined as in Fig. 13, and the functions are given by:

\[
A_\alpha = \frac{C_{cp}}{C_o} \left( 1 - \frac{\Delta T_N^\alpha}{C_p (m_L^\beta - m_o^\alpha)} \right) \quad \text{and} \quad A_\beta = \frac{C_{cp}}{C_o} \left( 1 - \frac{\Delta T_N^\beta}{C_p (m_L^\beta - m_o^\beta)} \right)
\]

\[ \lambda = \lambda_\alpha + \lambda_\beta = \frac{D_L}{V} (\ln A_\alpha^{1/2_k} + \ln A_\beta^{1/2_k}) \] ..................................(12)

The periodicity of the layers can then be written as:

\[ \lambda = \alpha_\lambda + \beta_\lambda = \frac{D_L}{V} (\ln A_\alpha^{1/2_k} + \ln A_\beta^{1/2_k}) \] ..................................(13)

From the analysis of the last two equations it is apparent that the thickness of the layers and their periodicity scales inversely with velocity for a given composition. It is also clear that the nucleation undercooling of the two phases plays a significant role in establishing the length scale of the layers.

5. Computational Modeling of the Peritectic and Dendritic Solidification

Deterministic modeling of dendrite growth is now a mature area of research. Yet, the published deterministic models for peritectic solidification are few. Zou and Tseng approached peritectic solidification of carbon steel assuming binary system and peritectic reaction mechanism. They included dendrite nucleation and growth in their macro-transport/transformation-kinetics model. The empirical law proposed by Jacobi and Schwefel correlating
austenite grain diameter to cooling rate was used as nucleation law. Apparently, the same relationship was used to calculate the number of δ grains. Little validation was presented for the liquid/solid transformation (no cooling curves). Data were given for dendrite arm spacing (but no explanation on how it was calculated), and on dendrite grain size, that should match experimental data anyway, since they serve as an input in the model.

Recent developments in solidification modeling have allowed the direct output and visualization of the phase morphology resulting from solidification. Of particular interest to this topic is the computational modeling of peritectic and dendritic phases through the phase field (PF) or the cellular automaton (CA) methods. Both methods have been demonstrated to be suitable for the problem at hand. While the PF method is more rigorous, it is computational intensive and as such relegated for the time being to the role of a tool for scientific investigation. Initially used to model dendrite solidification (see for example Ref. 31), its applicability to the modeling of peritectic solidification has also been established.32,33)

An example of dendrite growth simulation through the PF method is given in Fig. 14.34) At low nucleation rate there is enough space to develop a full dendritic morphology. Similar effects of nucleation rate and undercooling were demonstrated through the CA technique.35,36)

Tiaden37) simulated the microstructure evolution during peritectic solidification of Fe–C alloys using a multiphase field approach. It was assumed that the process is non-equilibrium, diffusion controlled. Three phases, liquid, ferrite and austenite, were considered, and phase fields were defined for each phase. The phase field model was coupled with a solute diffusion model. An example of the calculation of the growth of four ferrite particles during constant cooling of a Fe–C alloy is shown in Fig. 15. Anisotropy was not considered. Below the peritectic temperature single nuclei were placed on the four interfaces. It is seen that the peritectic γ grows around the primary δ by simultaneous consumption of both ferrite and liquid. The peritectic reaction is the fastest growth mechanism because at the peritectic temperature the carbon concentration in γ is higher than that in δ but smaller than that of the liquid. Thus the fastest growth will be where liquid and ferrite can directly react. Thus, the austenite grows around the ferrite.

Results of simulation of directional solidification under constant thermal gradient are shown in Fig. 16. The δ-dendrite produced by morphological instability growth in the liquid until the peritectic temperature is reached. At this temperature, some random nucleation of γ was imposed on the system, and then austenite continued to grow on the undercooled dendrite consuming both the ferrite and the liq-
uid. This model was later expanded to ternary systems (see Fig. 17). The CA method is more suited for engineering applications, but needs to overcome skepticism over its quantitative capabilities. As for the PF method, its suitability for modeling the evolution of the peritectic structure has been demonstrated. Indeed, Zhu and Hong have developed a CA method that can simulate microstructure evolution during eutectic and peritectic solidification. Their model, considers nucleation, growth and crystallographic orientation, and is coupled with the curvature and solute redistribution both in liquid and solid phase during solidification. The authors recognize model limitations such as cell size and dendrite orientation dependency. Nevertheless, they claim that the model is capable of predicting realistically complex nucleation and growth in both eutectic and peritectic systems.

The Zhu and Hong model was applied to simulate the peritectic transformation of a Fe–0.3mass%C alloy. The cooling rate and peritectic undercooling were chosen to be 10 K/s and 1 K, respectively. Other physical parameters were taken from Ref. 37. The simulation results are presented in Fig. 18. As the temperature decreases under the liquidus, primary α dendrites grow from the melt (Fig. 18(a)). When the temperature is below the peritectic temperature, β crystals nucleate at the α/liquid interface, followed by the growth of β phase around the primary α phase, since β can grow fastest where liquid and α have direct contact. The primary α phase is soon isolated from the liquid by the β phase (Figs. 18(b) and 18(c)). As the peritectic transformation proceeds, the thickness of the β layer increases by consuming both the liquid and a phase (Fig. 18(d)).

As noted by the authors, while randomization was used to generate the crystallographic orientations of dendritic nuclei, the resulted dendrites are parallel to the grid or oriented at 45 degrees. This is caused by the growth algorithm which adopts a neighborhood configuration including 8 neighbors.

Significant progress has recently been made in eliminating the mesh dependency of early CA models. Beltran-Sanchez and Stefanescu developed a quantitative model for simulation of dendrite growth controlled by solutal effects in the low Péclet regime. The model, based on CA concepts but using virtual tracking of the sharp S/L interface, produces realistic pictures of dendrite growth supported by validation. An example is provided in Fig. 19. The new model proposed a solution for the problem of mesh anisotropy valid for any grain orientation. The solution includes new methods for calculation of SL interface curvature, normal velocity, increment of the solid fraction, and trapping rules for interface cells. It also introduces a technique to minimize the mesh anisotropy by spreading the arc length of the SL interface over more than one cell, allowing...
the model to simulate dendrites growing at an arbitrary crystallographic orientation.

6. Concluding Remarks

While the progress in the understanding of the physics of peritectic solidification and its computational modeling is spectacular, it cannot be claimed that this research subject was brought to closure. Unresolved issues remain, mostly connected to the general approach in modeling of dendrite growth, which has not yet reached a satisfactory solution when quantitative results are expected.

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