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The existence of intragranular ferrite plates and nucleating inclusions in the heat affected zone of X-60 pipe steel

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In order to improve the heat affected zone (HAZ) toughness of X-60 pipe steel, we have applied intragranular ferrite plate (IFP) technology. The characteristic of IFP is the appearance of fine ferrite plates inside the original austenite grains. By means of suitable Re, Zr and Ti additions at high initial oxygen potentials, and good control of the peak temperature and the cooling rate during welding simulation, one can obtain IFP contents over 50 vol% with a resultant increase in the toughness from 55–160 J. It was found that the inclusions that were most effective in nucleating the IFP were deformable complex silicates which either entrap Re, Zr and Ti oxides or contain these elements. The greater the number of the evenly distributed and effectively nucleating inclusions, the greater the IFP content, and the finer the microstructure of the HAZ, and the greater the relevant toughness.

Generally, these silicates behave as fine spheres along a line. The present authors show that these fine spheres result from the remelting of the shuttle-like silicates due to heating in the process of welding simulation. These silicates contain a high sulfur capacity and thus MnS deposits are often observed on the periphery of the silicates. The IFP was shown to be directly rooted in the Mn depletion zone which is located beside the MnS deposits.

1. Introduction

Recently, considerable attention has been focused on intragranular ferrite plate (IFP or IGF) technology [1, 2]. This technology has produced a significant improvement in the toughness values of weld heat affected zones (HAZ) and free quench-tempered steel. The IFP process is characterized by the formation of fine ferrite plates inside the original austenite grains. This paper investigates the relationship between IFP formation and its function on the amelioration of HAZ toughness in X-60 pipe steel which is a major product of the Bao Shan Company. The compositional standard of this steel is listed in Table 1.

2. The experimental procedure

The samples are of 18 heats. A 50 kg induction furnace was used to carry out the experiments. When the bath temperature approached the range of 1880–1920 K, the initial oxygen level was controlled by predeoxidation and a solid sensor. If a high initial oxygen level was needed, only FeMn was added during predeoxidation. Then the Re, Zr and Ti were added according to a procedure designed on an orthogonal principle. The steel composition was then finally adjusted, but the bath was not killed with Al and Ca in this experiment.

Representative samples were taken from the lower part of the 20 kg ingot. Samples 12 mm in diameter and 100 mm in length used in the welding simulation were prepared by machine turning rods of 25 mm diameter, which were the forged products of 20 kg ingots.

A FIH 30S testing machine was used in the welding simulation. In the middle of the sample the direct heated zone with a size of less than 10 mm in length was set in the middle of the sample. During the heating stage it was heated up to 1650–1710 K. Above 1170 K, the cooling rate was 1 Ks^{-1} and between 1170–670 K, it was 5 Ks^{-1}.

After the welding simulation test, a V-type notch was made at the direct heated zone for further measurements on the toughness at room temperature. The microstructure of these broken samples were examined by use of optical microscopy, quantitative metallography, scanning electron microscopy (SEM) as well as energy dispersive X-ray spectroscopy (EDX). In addition, before welding simulation the forged samples were also observed using optical microscopy and SEM.

The instruments used were a Neophot-21 optical microscope, a Quantiment-500 imagine analysis system, an S-500 scanning electron microscope equipped with a 9100 EDX attachment.

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TABLE 1 The composition of the investigated X60 pipe steel

	C	Si	Mn	P	S	Nb	V	T.Al	Ca	Fe
%	0.066 / 0.105	0.305 / 0.405	1.30 / 1.50	< 0.015	< 0.005	0.035 0.045	0.045 / 0.055	0.015 / 0.045	0.0015 / 0.0045	bal.

For examination of the microstructure, nital was used to etch the samples. The IFP content was measured by use of the point counting method over more than 1000 points belonging to 10 visual fields.

3. Results and discussions
3.1. The improvement of the HAZ toughness

Excluding sample number 10, in which no Re, Zr or Ti was added, the HAZ toughness of the other samples can be divided into two categories. The average absorbed energy of the sample during impact testing at room temperature is used as a toughness index in this work. This index of the first group is 45 J on average. A common characteristic of this group is that the IFP content is below 20 vol%. On the other hand the second group has an IFP content of more than 40 vol%. The toughness index of this group is over 160 J on average, with a maximum value of 220 J.

Fig. 1 shows the relationship between the IFP content and the average absorbed energy. It is clear that to achieve an energy higher than 130 J then the IFP content should be greater than 50 vol%.

It is a generally accepted point of view that the finer the microstructure, the better the overall performance of the steel. Comparing Figs 2 and 3, the different microstructures of the two groups can be easily distingusihed. That of the first group is shown in Fig. 2. The developed ferrite distributes as a network along the original austenite boundary and then grows from there into the grain. Fig. 3, however, displays another feature in that much of the ferrite appears as plates inside the grains. The IFP content of this sample is 73 vol%. Thus the HAZ toughness is considerably improved.

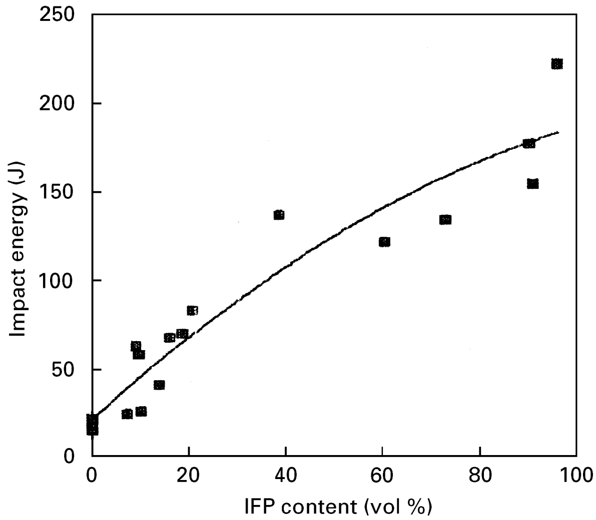

Figure 1 The relationship of the IFP content and the average absorbed energy of the sample during impact testing.

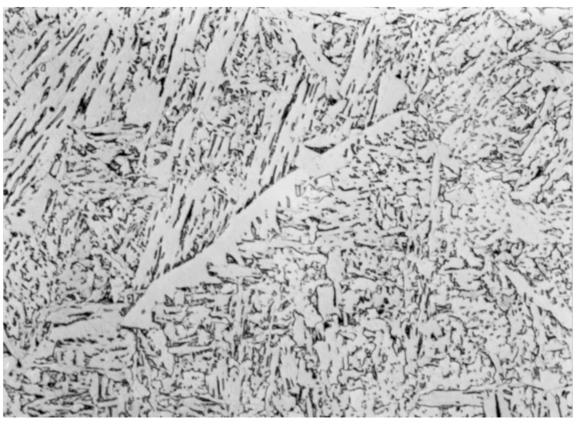

Figure 2 The HAZ containing either no or only a little IFP. Magnification × 260.

3.2. The formation of IFP

In what way, does the ferrite change its formation behaviour? Fig. 4 clearly shows that the "nucleating inclusions" are located at the intersection of the ferrite plates. Fig. 5 indicates that the distribution density of the inclusion should be more than 40 per mm^2 if the IFP content is required to be over 70 vol%. This density was measured with a Quantiment-500 image analyser. Undoubtedly, these two figures prove that the formation of IFP is facilitated by the "nucleating inclusion".

The effectiveness of various inclusions for nucleating IFP was found to be not quite the same. Essentially, the most effective inclusion is a heterogeneous complexed Mn-silicate, in which Re, Zr, Ti oxides were entrapped. Fig. 6 shows their SEM features, and a thin layer of MnS deposited from the silicate can be clearly observed.

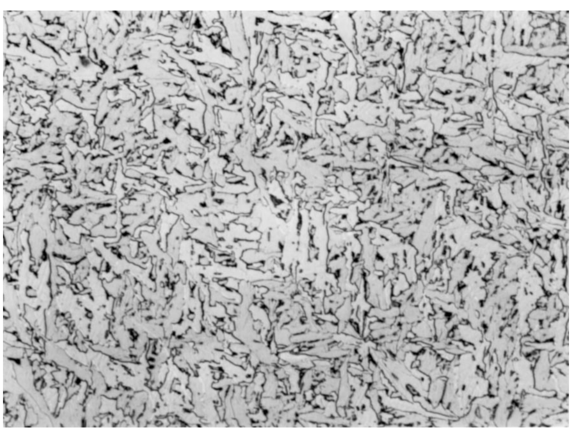

Figure 3 The IFP characteristics. Magnification × 260.

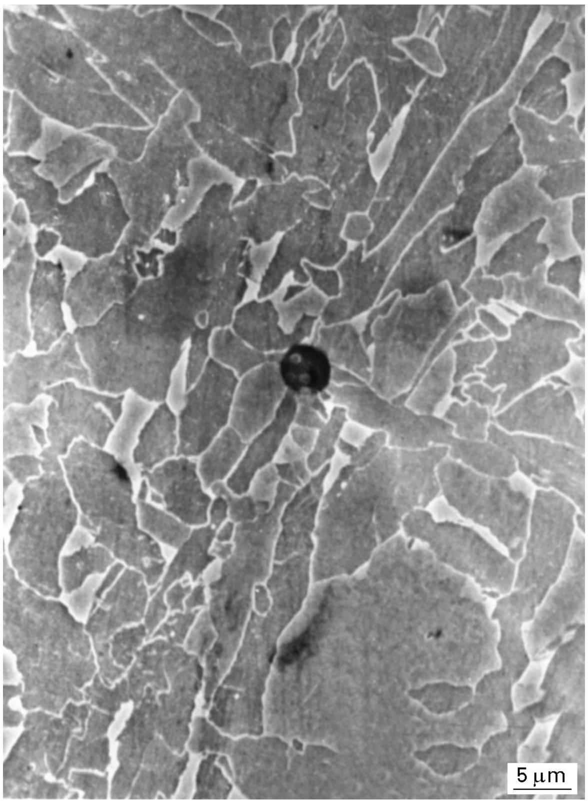

Figure 4 The effect of the inclusions on the nucleation of IFP.

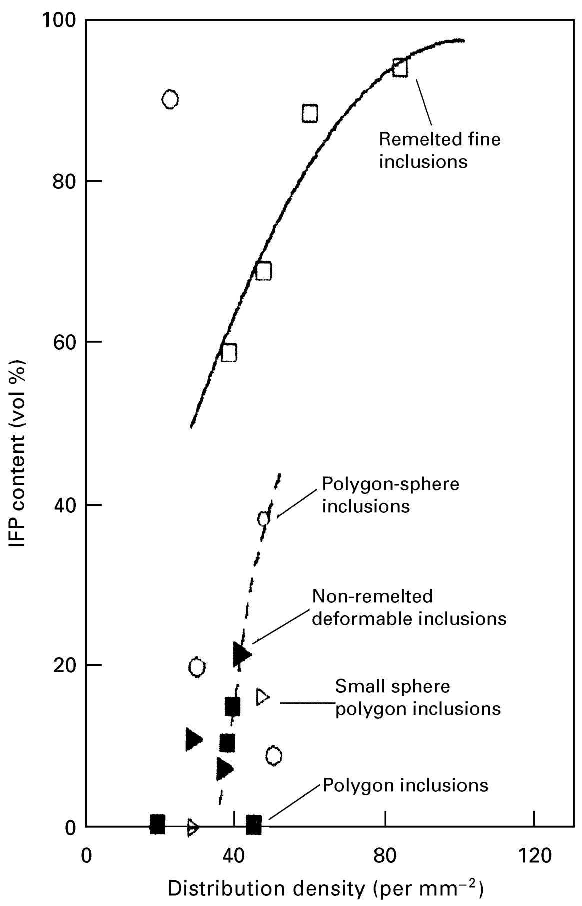

Figure 5 The relationship of the inclusion distribution density versus IFP content.

It was noticed by the present authors that these inclusions existed as fine spheres arranged in a line. Nevertheless, the deformable inclusions should behave as a sphere in the casting state, and after rolling or forging they should attain an extended shuttle-like shape. Fig. 7 displays an example of the shape modification process from shuttle-like to fine spheres. Certainly, the temperature along the length of the sample during welding simulation is continuously varied. Hence, it is possible to show the shape modification of the inclusion as a function of temperature gradient by observing the sample along its length. According to Fig. 7 at a location far from the directly heated zone during the process of welding simulation the silicates have a shuttle-like appearance. Approaching the directly heated zone the silicates are gradually remelted and again become sphere-like particles.

It was also found that some homogeneous Manganese silicates containing Re, Zr, Ti also exhibited effectiveness in the nucleation of IFP. The polygon-sphere like particles were found to be less effective in nucleating IFP. As a matter of fact, they are complex Re, Zr, Ti oxides including a small silica and MnS content. The brittle inclusions that generally are seen in the microscope as crushed pieces that are distributed as chains do not have any effect on the IFP nucleation.

3.3. The deoxidation

As may be seen in Fig. 6, the most effective "nucleating inclusion" is the heterogeneous complex consisting of silicate entrapped Re, Zr and Ti oxides. Also a MnS deposition can often be found on the silicate periphery. In fact, these components emerge under different conditions. According to thermodynamic arguments Re, Zr and Ti oxides form at once, when the alloys concerned are added to the heat. During steel solidification these oxides act as the nuclei for the separation of the main part of the silicate. The last one to be deposited from the silicates is MnS under a much lower temperature.

To promote the formation of IFP, some key points of the steel deoxidation procedure have to be adjusted. Fig. 5 shows it is necessary to have a high density of fine inclusion to promote IFP formation. Therefore, contrary to the popular deoxidation concept, the alloy whose oxide is the heaviest and has a tendency to evenly separate as fine particles should be selected. It is important to add the alloys into a bath with a high oxygen potential level. According to Fig. 8 (a and b) this level should be 200 p.p.m.

To make the silicate easily deformable and remeltable, Fig. 8 (a and b) indicates there is an optimum addition of these alloys. In the opinion of the present authors, it is advantageous to add 200 p.p.m. of Ti, 50 p.p.m. of Zr and 50 p.p.m. of Re. On the other hand, this will also create silicates with a higher sulfur capacity. In fact, the direct nuclei for IFP is just MnS. After MnS deposition, it grows by the consumption of the Mn in its surroundings. Thus, beside it a Mn depleted zone appears, the IFP is facilitated by this zone, while, the even distribution of MnS is provided by the previously even separation of the Re, Zr and Ti [3] oxides.

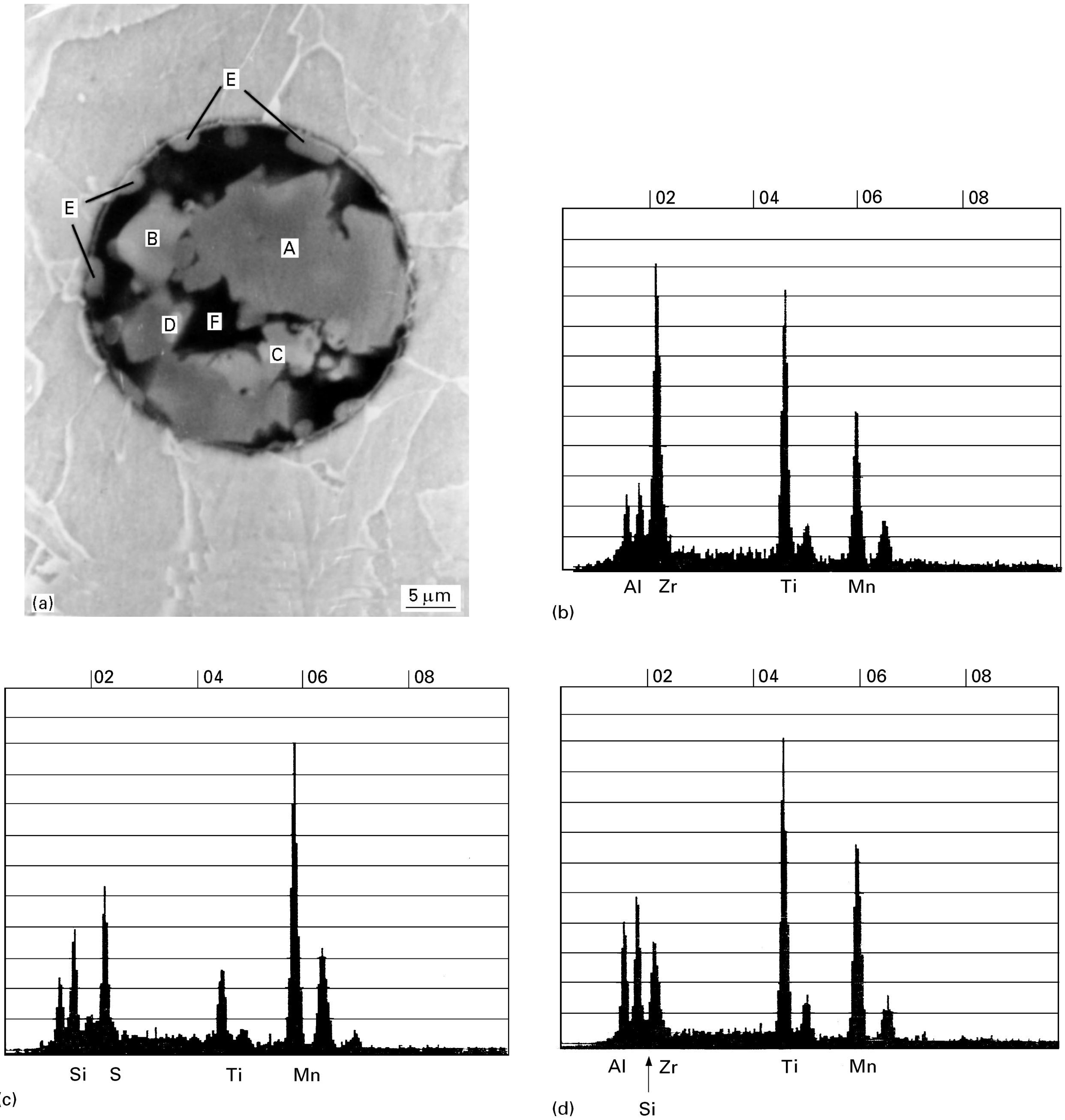

Figure 6 (a) The SEM micrograph of the heterogeneous nucleating inclusion and (b) the composition of point B which is similar to points A and C, (c) the composition of point E and (d) the composition of the matrix point F, of the inclusion. The composition of point D is similar to point F.

Tomita *et al.* claim to have measured a sulfur content of 30–40 p.p.m. [4]. This is in agreement with this investigation. For nitrogen, Tomita *et al.* indicated that it was advantageous to have a N content lower than 20 p.p.m., if Ti oxides are chosen to play the role of ferrite nuclei in HAZ [4]. This, however, is not in agreement with the present research. In this investigation, the N content reaches 50–140 p.p.m., and no evidence concerning TiN particles playing a direct role in the nucleation of IFP was found. Notwithstanding this point it is certain that this higher nitrogen content might be the reason for an unstable yield of Ti addition.

Long *et al.* have reported the results of experiments carried out under the conditions of 40 p.p.m. initial oxygen potential and 50–100 p.p.m. sulfur content. They have emphasized the function of lattice disregistry on the formation of IFP [5]. The IFP indued by TiN, in the viewpoints of some researchers, solely results from a small lattice disregistry. As previously mentioned, this experiment did not find the IFP being directly generated by TiN, so perhaps, the effectiveness of Mn depleted zones is superior than that of lattice disregistry.

4. Conclusions

The IFP process can be promoted by adjusting the deoxidation step to ensure a high density of fine, evenly distributed heterogeneous composite silicates entrapping Re, Zr and Ti oxides. These inclusions are deformable and gain a higher sulfur capacity. During welding simulation these deformed inclusions were shown to remelt and then again exhibit sphere-like

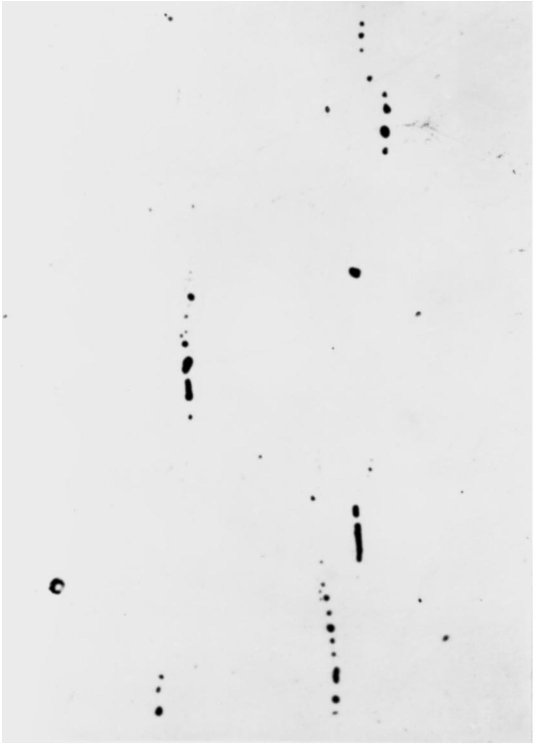

Figure 7 The remelting process of the inclusion during the welding simulation test. Magnification $\times 320$.

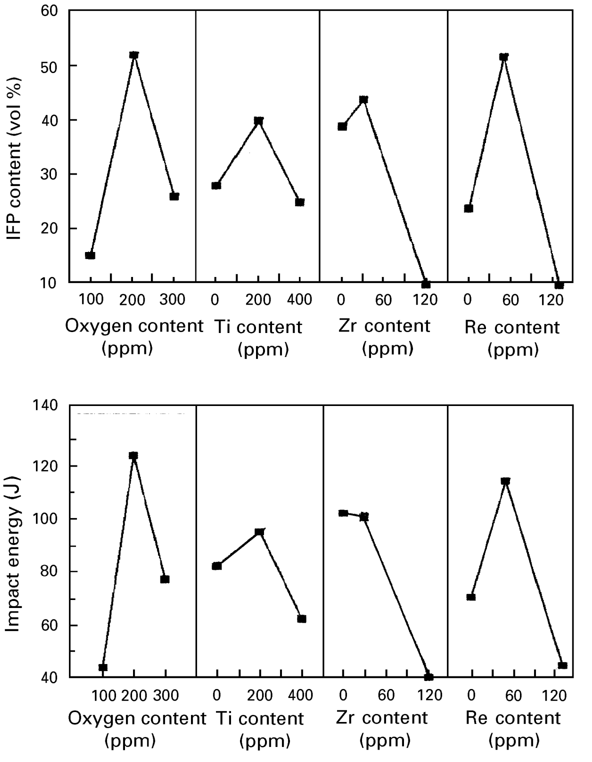

Figure 8 The influence of the deoxidation scheme on the IFP content and the average absorbed impact energy.

particles. In the lower temperature zone, MnS is deposited from the silicates, which leads to the formation of a Mn depleted zone. The ferrite plates inside the austenite grain nucleate and grow in this zone.

The greater the number of the effective nucleating inclusions, the greater the IFP content, and the finer the microstructure of the HAZ, and thus the greater is the relevant toughness.

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