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Some microstructural features of the welds in butt-welded polyethylene and polybutene-1 pipes

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Microstructural features characterizing butt welds in polyethylene and polybutene-1 pipes have been studied using scanning electron and transmission optical microscopy. The microstructures are explained on the basis of temperature gradients measured in the region of the welds and the flow of molten material during welding.

1. Introduction

In any welding process for plastics, heat is applied to melt the surfaces to be joined and these surfaces are then pushed together and allowed to cool. It is unlikely therefore that any microstructure in the weld will be the same as that of the rest of the material; large thermal gradients will have been caused, there is the possibility of row nucleation at the joined surfaces, a certain amount of flow must have occurred and different parts of the weld will cool at different rates. It is obviously essential to understand the microstructures to discover reasons for service failures, yet little has been published [1, 2] in spite of the widespread and growing use of plastic pipes. The present paper shows how the microstructures can be revealed for polyethylene and polybutene-1 butt welds and offers an explanation for these microstructures.

2. Experimental

In the butt-welding process, the pipe ends to be joined are cut so that they are smooth and plane and wiped with an acetone swab to remove any grease. The ends are brought up to a Teflon-covered heating plate and heated for a short time. The heating plate is then removed and the two halves pushed together and held in contact. A small bead forms on the inside and outside surfaces (see the section shown in Fig. 1) thereby indicating overall melting and some flow at the surfaces joined. (An experienced welder will tell the quality of the weld from the size and shape of the bead.) In our experiments a Bielomatik welding machine (Model HV493) supplied by Bielomatik Lenze Co, West Germany, was used.

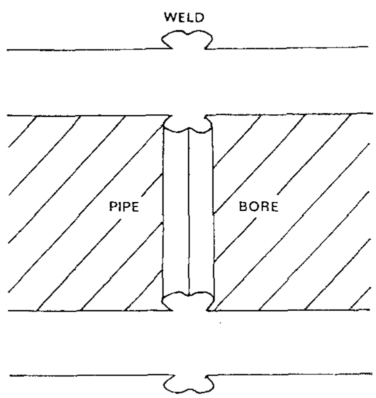

Figure 1 Section through a butt-welded pipe.

The quality of each weld was evaluated in terms of a welding parameter "*f*" defined by Menges and Zöhren [1] as the ratio of the yield stress of the welded material over that of the non-welded material. For a good weld, this ratio was equal to or greater than unity. In these tests [3], longitudinal sections across the weld were sawn from the pipes and the weld beads machined off. Tensile specimens with a 25.4 mm (1 in.) gauge length were cut out using a router and pulled in an Instron tensile testing machine at a strain rate of 0.83 mm sec^{-1}.

We have found the following optimum conditions for welding Plastronga polyethylene pipes (outside diameter in the range 60.1 to 60.6 mm, wall thickness 6.6 to 7.3 mm, density about 0.96×10^3 kg m^{-3}) kindly supplied by Yorkshire Imperial Plastics of Leeds, UK. The temperature at the surface of the heating plate should be 235°C and the pipe ends should be brought up to the heating plate and held there for 20 sec with just sufficient pressure to ensure complete surface contact between heating plate and pipe end. After 20 sec the pressure should be released but the pipe still heated for a further 35 sec. The two ends of the pipe are then pushed together using a pressure of about 0.1 MN m^{-2} and this pressure maintained for at least 180 sec. For the polybutene-1 pipe (outside diameter 64 mm, wall thickness about 3.3 mm, density about 0.92×10^3 kg m^{-3}) supplied by Polva-Nederland of Holland the optimum conditions were slightly different. The temperature at the surface of the heating plate was 245°C and the pipes were heated under slight pressure for 15 sec and then for 22 sec with no pressure applied. The two ends were pushed together under a pressure of 0.15 MN m^{-2} and held for at least 180 sec.

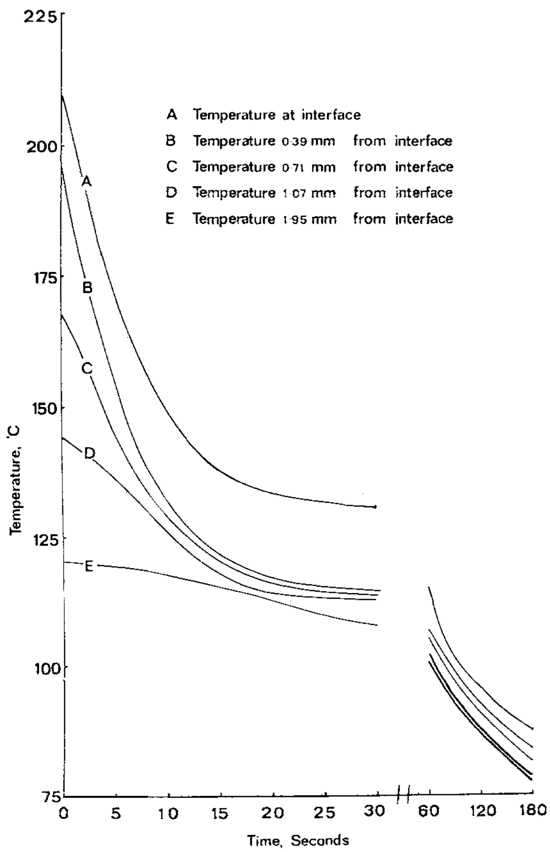

Figure 2 Graph showing the variation of temperature with time at different distances from the interface.

Similar welding conditions have been reported by other workers [1, 2].

There is a region in the neighbourhood of each weld where the microstructure has been altered by the welding process. The extent of this region can be estimated from Fig. 2 which shows the fall in temperature with time in the centre of the pipe wall and at different distances from the contact point of the two surfaces. These results were obtained in the following way. A spiral groove of depth about 1 mm and with a pitch of 4 mm was cut in the outer surface of the pipe and holes were drilled at different points along the groove half-way through the wall thickness. The holes were of such a diameter to produce an interference fit with the chromel-alumel thermocouple beads. Thermocouples were inserted at different distances from the interface and connected to a UV Oscillograph and the variation in temperature with time recorded continuously for each. Fig. 2 shows that there is a steep temperature gradient in the region of the weld. Fig. 3 shows the maximum temperatures reached at different distances from the weld and it can be seen that for this particular pipe there is a region extending for about 2 mm on each side of the centre portion of the weld which has been heated above its melting point.

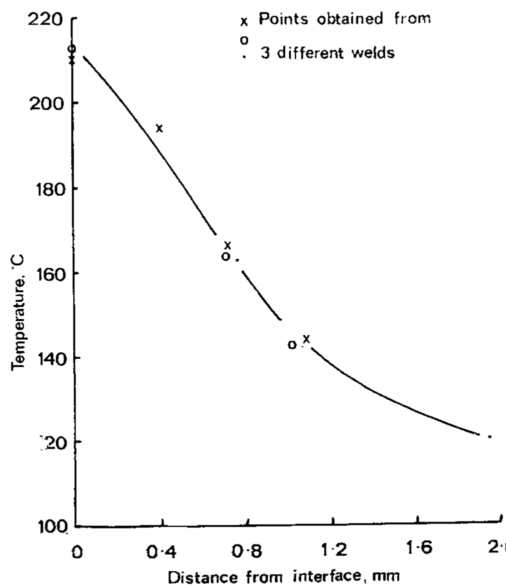

Figure 3 Graph showing the maximum temperatures reached at different distances from the interface.

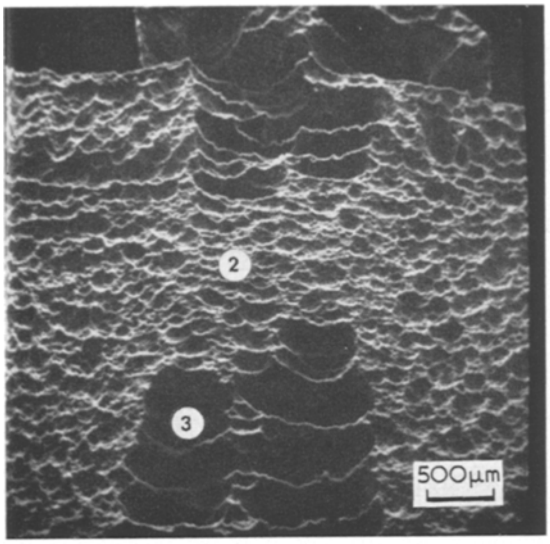

Figure 4 Polyethylene weld after etching for three days in chromic acid at 67°C. Magnification × 16. (Scale bar = 500 µm.)

Figure 5 Polyethylene weld after etching for 12 h in chromic acid at 67°C. Magnification × 17. (Scale bar = 500 µm.)

3. Results

3.1. Microstructural features in polyethylene welds

Armond and Atkinson [4] have discussed the use of 6M chromic acid as an etchant for polypropylene. They suggested that the carbon-carbon bond involving the methyl group is relatively easily oxidized and that such bonds are more accessible in amorphous regions than in crystalline regions. Hence chromic acid preferentially attacks amorphous regions which are washed away to reveal the crystalline structure. Polyethylene has no methyl links and is characterized by its stability to oxidative attack. For this material, therefore, long oxidation times would seem to be necessary. Fig. 4 is a Stereoscan micrograph of the surface of a specimen cut across the weld after treatment in a saturated solution of chromic oxide (CrO_3) in water at 67°C for three days. It can be seen that attack is very pronounced and that the welded region has been attacked much more than the rest of the pipe. An interesting feature is the sharpness of the weld boundary; it appears that there is a distinct microstructural change in the region of the weld and the weld structure does not extend gradually into neighbouring regions.

The fact that the region in the neighbourhood of the weld has been attacked more than the rest of the pipe suggests either that the welded region is more amorphous, or that a crystalline structure has been produced which is particularly susceptible to chromic acid attack. The lack of fine detail indicates that the material has been over-etched and further experiments showed that etching times between 6 and 12 h were sufficient. Fig. 5 shows that the most severe attack occurs in zone 3 (the numbers on the micrographs refer to the zones described in Fig. 10).

3.2. Microstructural features in polybutene-1 welds

Samples of polybutene-1 were also etched and

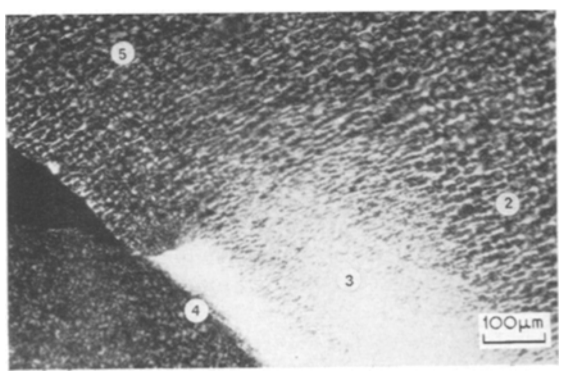

Figure 6 Microtome section of polybutene-1 weld showing the base material in the bottom left corner, a whitened region (zone 3) in the centre of the picture separated from the base material by a boundary layer (zone 4), zone 2 on the right of the picture and the bead (zone 5) at the top. Magnification × 82. (Scale bar = 100 µm).

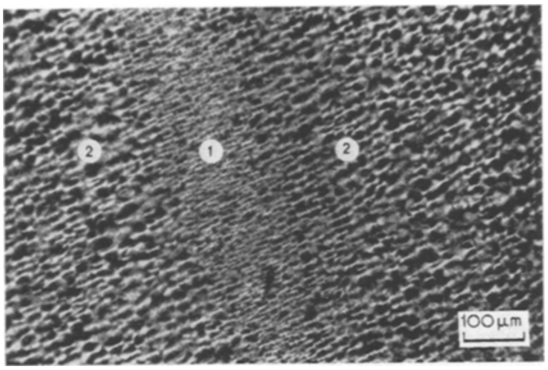

Figure 7 Microtome section of polybutene-1 weld showing the centre of the weld (zone 1) with zone 2 on either side. Magnification × 82. (Scale bar = 100 µm.)

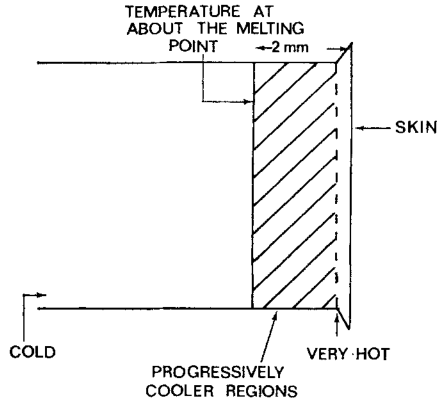

Figure 8 Diagram showing a section through the pipe wall during the second stage of welding.

viewed in the Stereoscan, but the electron beam caused surface etching and doming. Hence this technique was not pursued but thin sections were cut across the weld using a microtome and these sections examined in transmitted light with crossed polars using a Vickers M55 light microscope. The results (Figs. 6 and 7) show that the welded region can be divided into five zones. Similar zones have been found in polyethylene welds by Menges and Zöhren [1]. The base material has a uniform spherulitic appearance with spherulites approximately 20 µm in diameter (Fig. 6, left). In zone 4, which is very narrow, it has been suggested [1] that the welding pressure displaces the material and causes an orientation due to thermoelastic upsetting at the boundary layer of the "cold" material. Zone 3 consists of a fine crystallite structure and zone 2 a spheroidal structure. Fig. 7 shows zone 1. This zone marks the boundary between one half of the pipe and the other, but does not exist along the entire boundary line. Near the top and bottom of the pipe wall and in the bead it is only just possible to make out this boundary between the two halves.

4. Discussion

In the first stage of welding, the two pipe ends are held against the heating plate and a small lip forms. We have shown that, using optimum welding conditions, the temperature falls rapidly with distance from the heating plate and reaches the melting point about 2 mm away on each side. In the second stage, the heating plate is removed and there will be a short period before the pipe ends can be pushed together. During this stage a chilled skin containing crystallite nuclei will form at the pipe ends (see Fig. 8). In the final, joining stage, the pipe ends are pushed together until a bead of the required size and shape forms. The flow of material and the consequent temperature distribution are indicated in Fig. 9, whilst Fig. 10 shows the microstructures which would result from this temperature distribution.

First, let us consider Fig. 9. The boundary layer (4) will be at a temperature less than the melting point but greater than the softening point, so the layer will deform easily and produce a smooth boundary with the colder material. The thickness of the cooler layer (2) will be greatest in the centre of the pipe, where the material was furthest from the hot plate in stage one, and decrease towards the surfaces. Nucleation will occur where the cooler layer meets the boundary layer. The hottest material (3) will be pushed away from the centre of the pipe and into the beads. The thickness of the central skin (1) will be determined initially by the time interval when the heating plate is removed but before the pipe ends are pushed together. On contact, adhesion will occur and the central skin will remain almost stationary whilst hotter material flows past it. Near the surfaces the adjacent material (3) will be hot enough to melt part or all of the skin.

The resultant microstructures are shown in Fig. 10. Nucleation will occur all along the boundary (4); however, only near the surfaces will the adjacent material (3) be hot enough for there to be a thermal gradient large enough to induce columnar growth towards the centre line of the weld. The relatively cool material (2) will soon crystallize with a slightly elongated spherulitic microstructure due to the tempera-

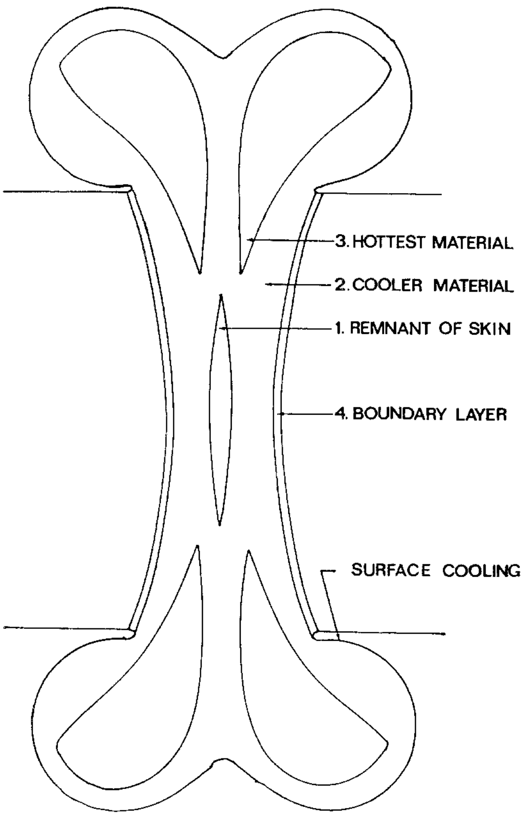

Figure 9 Diagram showing the temperature distribution in a section through the pipe wall during the final stage of welding.

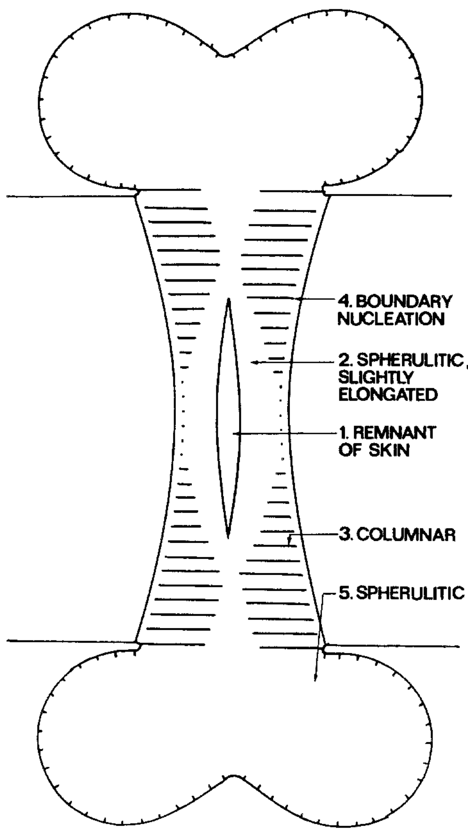

Figure 10 Diagram showing the expected microstructures in a section through the pipe wall after completion of the final stage of welding.

ture gradient (see Fig. 7). In the beads, the material (5) is hot and much flow has occurred; heat will be lost radially and solidification of the surface will occur. Thereafter the rate of cooling will fall and the remaining material will crystallize with a conventional, spherulitic structure. The microstructure of the skin (1) will be determined by the welding pressure used. At low pressures, the thickness of region 2 will be relatively large and the material adjacent to the skin will be relatively hot; hence the skin will melt, nuclei will be destroyed and subsequent slow cooling will produce relatively large spherulites. At somewhat higher welding pressures, the thickness of region 2 will be smaller, the material adjacent to the skin will be cooler, some nuclei in the skin will remain and some directed growth may occur from the skin into region 2 (see Fig. 7). If the welding pressure is high, region 2 will be very thin and the cool material adjacent to the skin will be near the melting temperature. Rapid cooling will produce a "fine-grained" structure of small spherulites in zone 1.

5. Conclusions

The following points emerge. First, there is a steep temperature gradient in the region of the weld. Second, the microstructures of both polyethylene and polybutene-1 welds consist of five different zones. In this context it is noteworthy that the columnar structure in polyethylene welds is revealed easily by chromic acid etching. We believe that this is due to the high concentrations of impurities in front of and between the columnar crystals. Third, these different zones which will characterize all butt welds of crystalline plastics can be explained on the basis of the flow of material at the weld and the consequent temperature variations.

We would now briefly like to consider possible weaknesses in this type of microstructure. Because of the impurities, a columnar structure will have weak boundaries longitudinally and

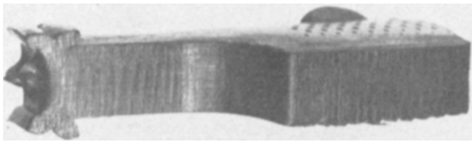

Figure 11 Fracture of a polyethylene weld caused by a tensile stress.

also laterally where the columnar crystals meet ordinary spherulites. Longitudinal weaknesses would assume importance in stress rupture tests where the application of an internal pressure would result in a hoop stress which would separate columnar crystals at their boundaries. However, probably of more importance is the lateral weakness for this weakness will always be exacerbated by the notch between the weld bead and the pipe. In practice the weld bead will always be left on and we have found that failure in tension will then occur as shown in Fig. 11. We are at present investigating such failures in detail.

In conclusion, we feel that if directional solidification produces markedly deleterious effects it may be necessary to reconsider the conventional butt-welding technique. We are considering the effect of using a heating plug in conjunction with an external heating coil.

Acknowledgement

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