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Ancient Monuments Laboratory
Report 35/95

METALLOGRAPHIC EXAMINATION OF
MISCELLANEOUS IRON OBJECTS
1994

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Summary

This report provides details of a number of (unassociated) ferrous objects examined at the Ancient Monuments Laboratory during 1994. These comprised an unusual, internally oxidised cast iron fragment from Windsor Castle, Berkshire, a casting spill of cast iron from Tilbury Fort, Essex and parts of a wrought iron gate and railings from Garrison Church, Portsmouth.

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Cast Iron Object from Windsor Castle Round Tower

Macro Examination

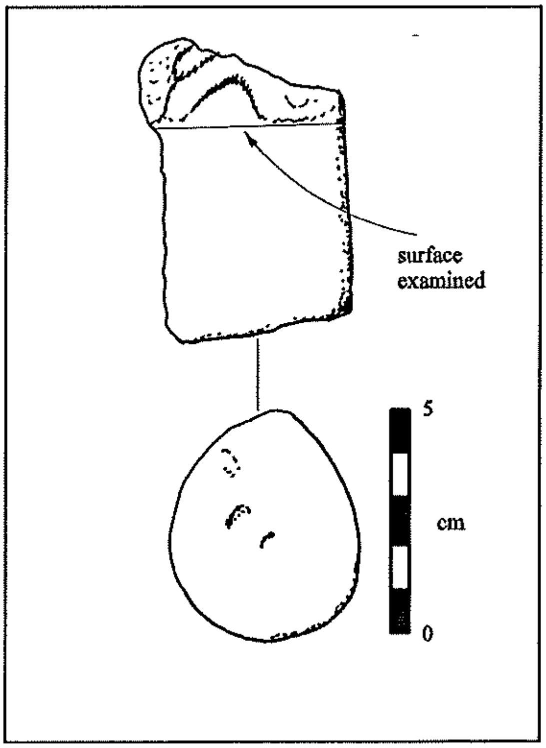

Figure1 Windsor Castle cast iron object

Find No. 13018, was an iron object (Fig. 1) of approximately cylindrical shape with a length of 50mm, diameter of 40mm and weight of 283g. The artefact was recovered from context 7101 during the excavation of the Round Tower. Although originally classed as iron, the object showed unusual corrosion products; a hard, dense layer with a distinctly purple/maroon tinge. Qualitative X-ray fluorescence (XRF) analysis of the surface identified only iron and manganese, ruling out the possible presence of unusual alloying elements or surface treatments. Streak testing of the surface on a porcelain tile gave a red-brown colour, suggestive of hematite (Fe_2O_3), but the response to the magnet showed that at least some iron, or possibly magnetite (Fe_3O_4), lay beneath the surface. Repeated density measurements (5.4, 5.05 & 5.05) gave a mean value of 5.16 g cm^{-3}, which is much closer to the density of magnetite and hematite (both 5.2 g cm^{-3}) rather than pure metallic iron (7.9 g cm^{-3}) or cast iron (6.8 to 7.8 g cm^{-3} depending on structure). However, the remarkably regular shape of the object was not consistent with it being of natural mineralogical origins and the artefact was therefore sectioned for metallographic examination.

Metallographic Preparation

The exposed section of the object was prepared using standard metallographic techniques; grinding on successively finer abrasive papers then polishing with diamond impregnated cloths. The specimen was examined on a metallurgical microscope in both the "as polished" i.e., unetched condition and after etching in 2% nital (nitric acid in alcohol). A Shimadzu microhardness tester was used to determine the hardness of different phases within the metallographic structure.

Metallographic Structure

Viewed at low magnification in the **unetched** condition (Plate 1) frequent gas pores (5% by area) were observed in the structure together with what appeared to be very coarse graphite flakes (10%). However, at higher magnifications the "graphite flakes" were shown to have very ragged edges and to contain corrosion products. **Etching** in 2% nital (Plate2) revealed the grain structure of the metallic phases between the flakes/corrosion to be predominantly ferrite (75%) of grain size ASTM 6. The ferrite grains contained some spheroidised iron carbide and further irregularly shaped carbide (10%) was present at the ferrite grain boundaries.

Microhardness tests on the ferrite+carbide spheroidised regions showed considerable variation:

(ferrite + pearlite)	292.6 H_v
	324.6 H_v
50g load	236.5 H_v
10sec. load time	276.9 H_v
	220.6 H_v
Mean	270.2 H_v

Interpretation of the Structure

The metallographic structure is that of a grey cast iron which has been transformed at high temperature. Spheroidisation of pearlite indicates that this heating would have been prolonged, perhaps of the order of hundreds of hours at 650°C. Cyclical heating is also indicated by the corrosion within the metal where hot gasses have entered the artefact through the graphite cavities. The resultant volume increase would have set up stresses within the object which may have resulted in its failure. The function of the artefact is unclear, fracture surfaces at both ends suggest that this was only a fragment of a larger cast iron object. The severely overheated structure must derive from the use of the object, rather than its manufacture, and it may well have formed part of a furnace, domestic stove or fireplace furniture.

No dating evidence was provided with the object. Technologically it is extremely unlikely that objects in cast iron predate the blast furnace, of which the first British example dates from 1496. However, Continental European blast furnaces, and stückofen, which could also produce cast iron, did exist earlier. Occasional examples of "accidental" cast iron production, using the traditional bloomery process are known in this country as far back as the Roman period, though these are assumed to have resulted from mistakes in running the furnaces, as no attempts to utilise the material are recorded.

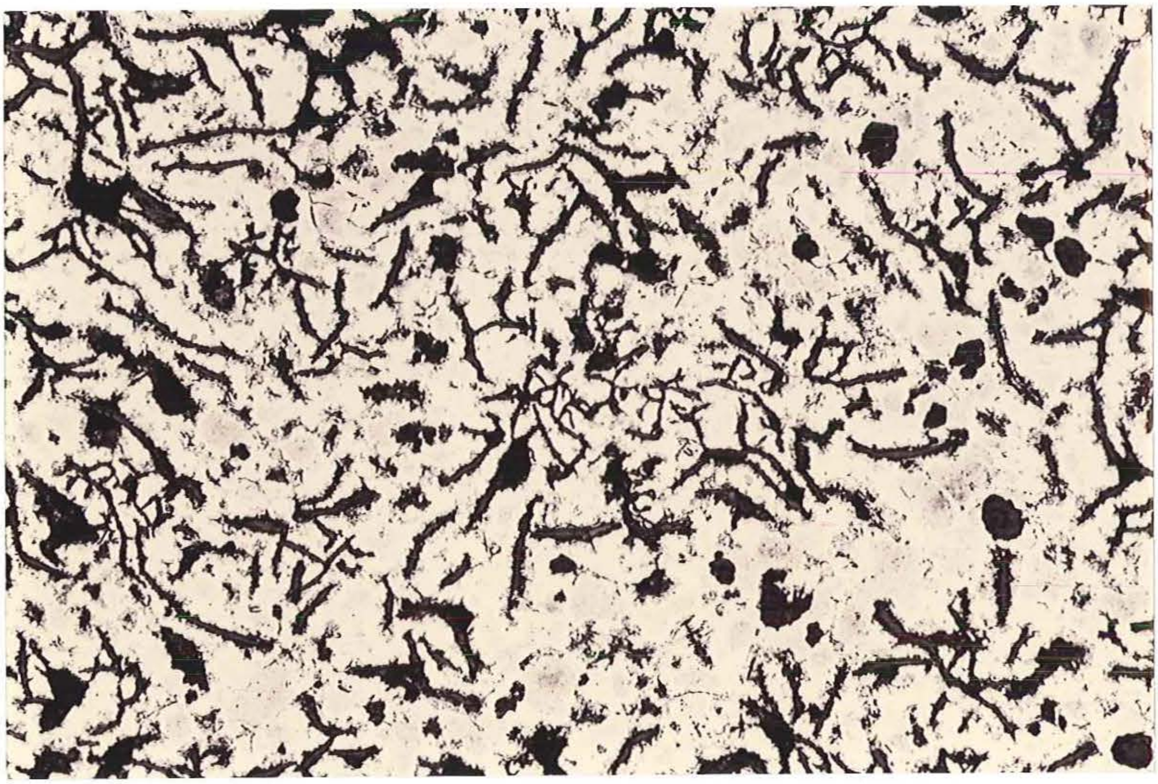

Plate1 Windsor Castle cast iron object Micrograph: unetched x100
Corrosion in voids left by graphite flakes

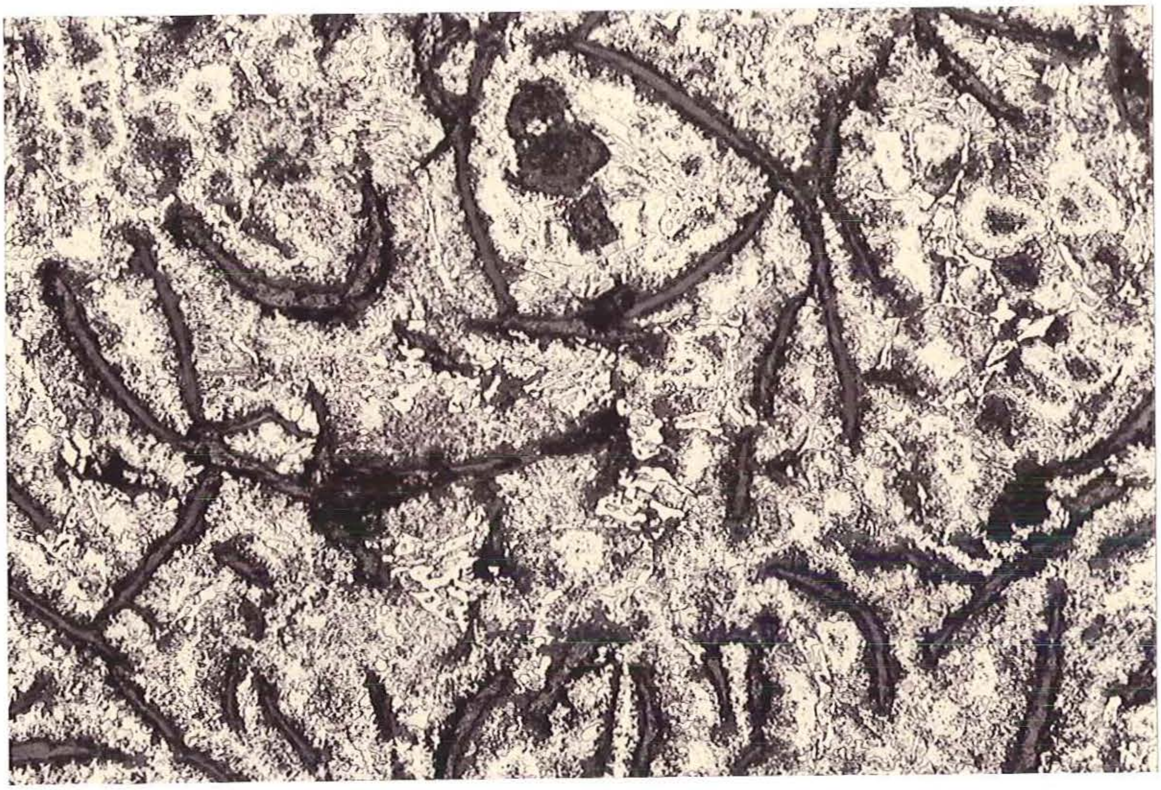

Plate2 Windsor Castle cast iron object Micrograph: etched in 2% nital x200
Ferrite with spheroidal and grain boundary iron carbide

Iron Casting Spill from Tilbury Fort, Essex

Macro Examination

An iron object (small find 443), from context 4200 at Tilbury Fort, originally thought to be part of a bloom, was received by the AM Lab. for identification. The object, (Fig. 2) was of approximately plano-convex shape had a maximum length of 80mm, width (at 90° to max. length) of 45mm, thickness of 13mm and weight of 135g. The upper surface was reasonably smooth whilst the lower was uneven, with lumps corresponding with the discrete light patches on the X-radiograph.

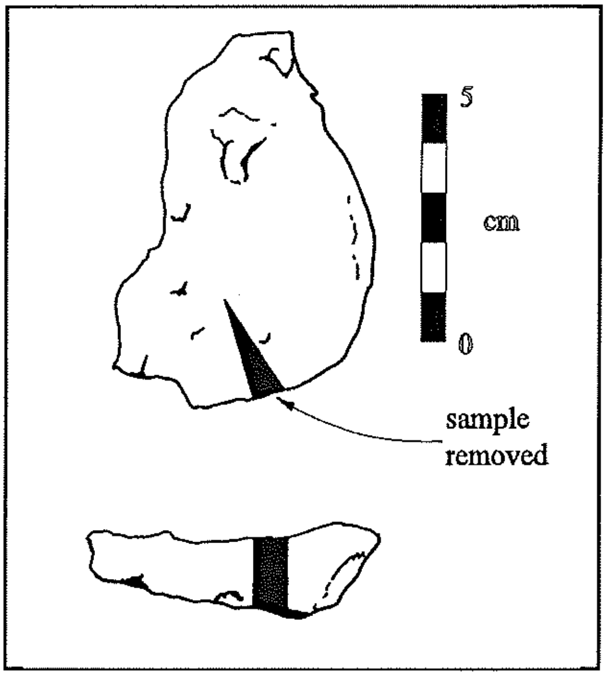

Figure2 Tilbury casting spill

Metallographic Preparation

A "V" shaped notch was cut from the object, mounted in phenolic resin and prepared using standard metallographic techniques; grinding on successively finer abrasive papers then polishing with diamond impregnated cloths. The specimen was examined on a metallurgical microscope in both the "as polished" i.e., unetched condition and after etching in 2% nital (nitric acid in alcohol). A Shimadzu microhardness tester was used to determine the hardness of different phases within the metallographic structure.

Plate3 Tilbury Fort iron casting spill. Micrograph: unetched x100
Coarse graphite flakes

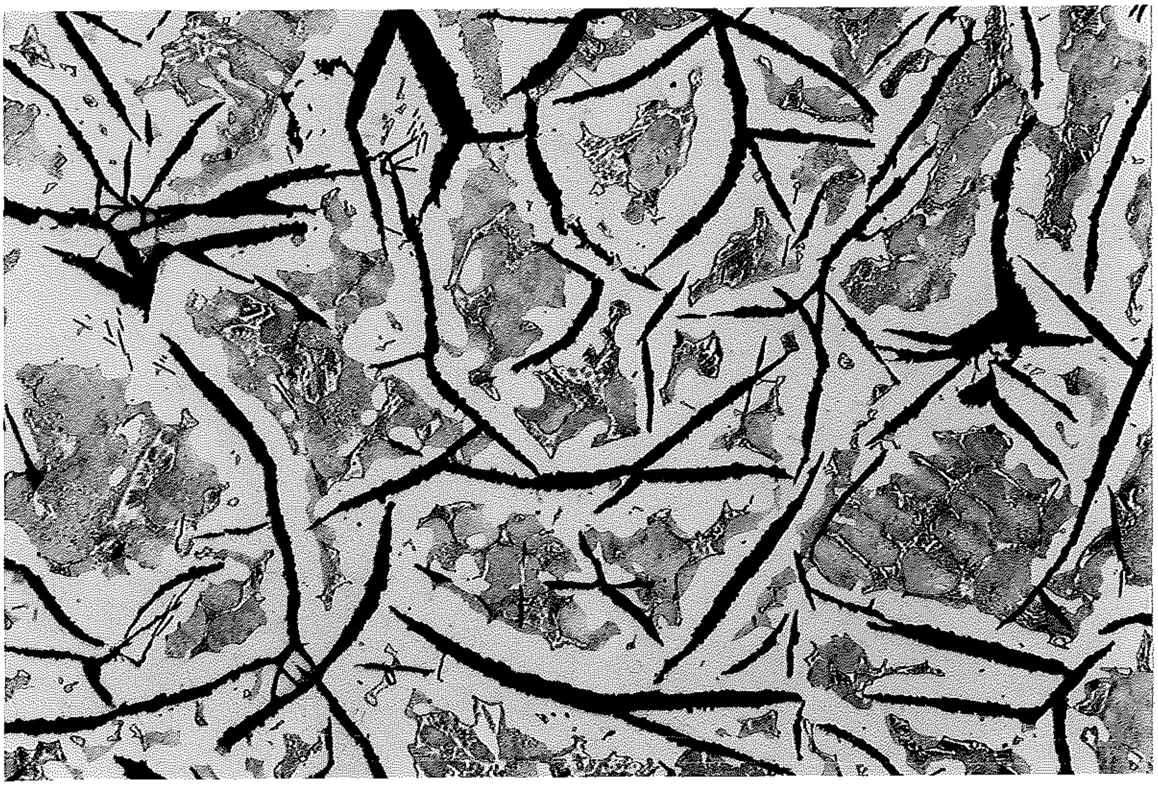

Plate4 Tilbury Fort iron casting spill. Micrograph: etched in 2% nital x100
Ferrite surrounds the graphite, phosphorus eutectic between the pearlite dendrites

Metallographic Structure

Viewing in the **unetched** condition (Plate 3) revealed coarse graphite flakes (10%). **Etching** in 2% nital (Plate 4) showed the graphite flakes to be surrounded by ferrite (40%) in the areas between these, very fine pearlite is present in dendritic form(40%) and between the dendrites can be seen the laminated structure of phosphorus eutectic.

Microhardness tests on three metal phases gave the following values (50g load, 10sec. load time):

ferrite	362.2 H_v	pearlite	508.8 H_v	phosphide	423.3 H_v
	231.8 H_v		441.0 H_v	eutectic	593.4 H_v
	309.8 H_v		340.6 H_v		726.1 H_v
	265.1 H_v		381.0 H_v		713.5 H_v
	292.6 H_v		385.9 H_v		766.3 H_v
mean	292.3 H_v		411.5 H_v		644.5 H_v

Interpretation of the Structure

Metallographic examination shows the metal to be common grey cast iron containing a small amount of phosphorus. The latter element is present in the form of phosphide eutectic, a laminated constituent comprising of ferritic iron, cementite (Fe_3C) and iron phosphide (Fe_3P). The presence of phosphorus is not unusual, the element is co-smelted with the iron from the ore, and its concentration would have been insufficient to cause significantly increased brittleness. There is no evidence of any further heat treatment to the metal. The morphology of the cast iron indicates that the find is not a deliberately manufactured object, but a waste product, probably a spill from the casting of iron.

The term "cast iron" is used for alloys of iron and carbon where the latter is greater than about 2%. The effect of the carbon is to reduce the melting temperature of the iron, from a maximum of 1535°C, for pure iron, to a minimum of 1130°C, at 4.3% carbon. Thus the casting of "cast iron" was possible at a much earlier date than pure irons or steels i.e. iron containing approximately 0.25 to 1% carbon. In England the casting of "cast iron" would have been possible from 1496 using liquid metal direct from the blast furnace in which the iron had been smelted or, after 1794, by using a cupola to remelt pigs of cast iron. Although instances are known where cast iron was, presumably inadvertently, produced from the earlier bloomery furnace this was probably a very unusual occurrence. A second property of cast iron is that, unlike the malleable products of the traditional bloomery furnace, cast iron is unworkable in the forge and little secondary work can be carried out on the material without machine tools.

Wrought Iron Gate from Garrison Church, Portsmouth

Introduction

Sections of corroded wrought iron railings and a gate from Garrison Church, Portsmouth were removed for conservation at English Heritage's Architectural Metalwork Conservation Studio. Prior to restoration The Ancient Monuments Laboratory was contacted to examine the ironwork. In particular it was hoped to determine whether either the installation of the gates, or any subsequent repairs relate to the known restoration of the church in 1868.

Macro Examination

Figure 3 shows a length of the square sectioned railings from the Church. Many of the uprights were highly corroded; the pattern of corrosion revealing the laminated structure of the metal. The more ornate gate is illustrated in Figure 4. Corrosion on this also showed, though to a lesser extent, the lamellar nature of the iron. In addition raised areas of weld metal were observed, joining the different components of the gate, the poor "as welded" finish of these was in contrast to the otherwise well-built gate, suggesting that they date from a later repair. It was also thought that two small triangular strengthening fillets, below the joint of the hinged upright and the top and bottom horizontal rails, were likely to be later additions.

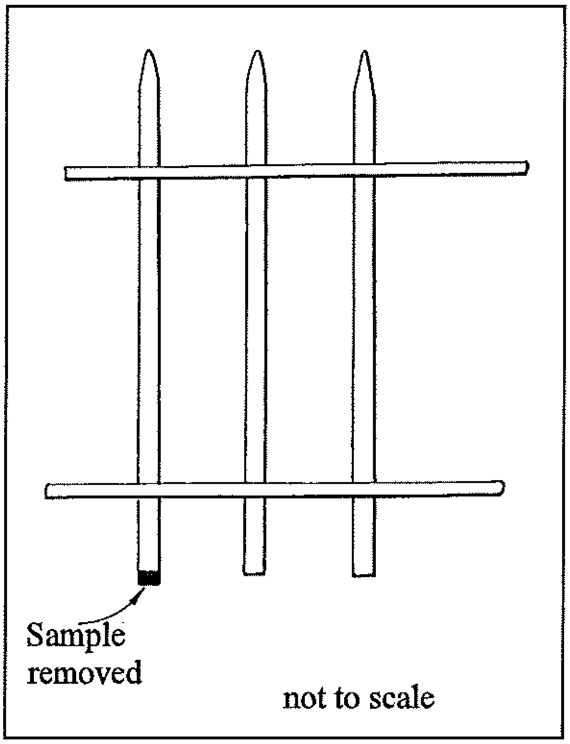

Figure 3 Garrison church railing

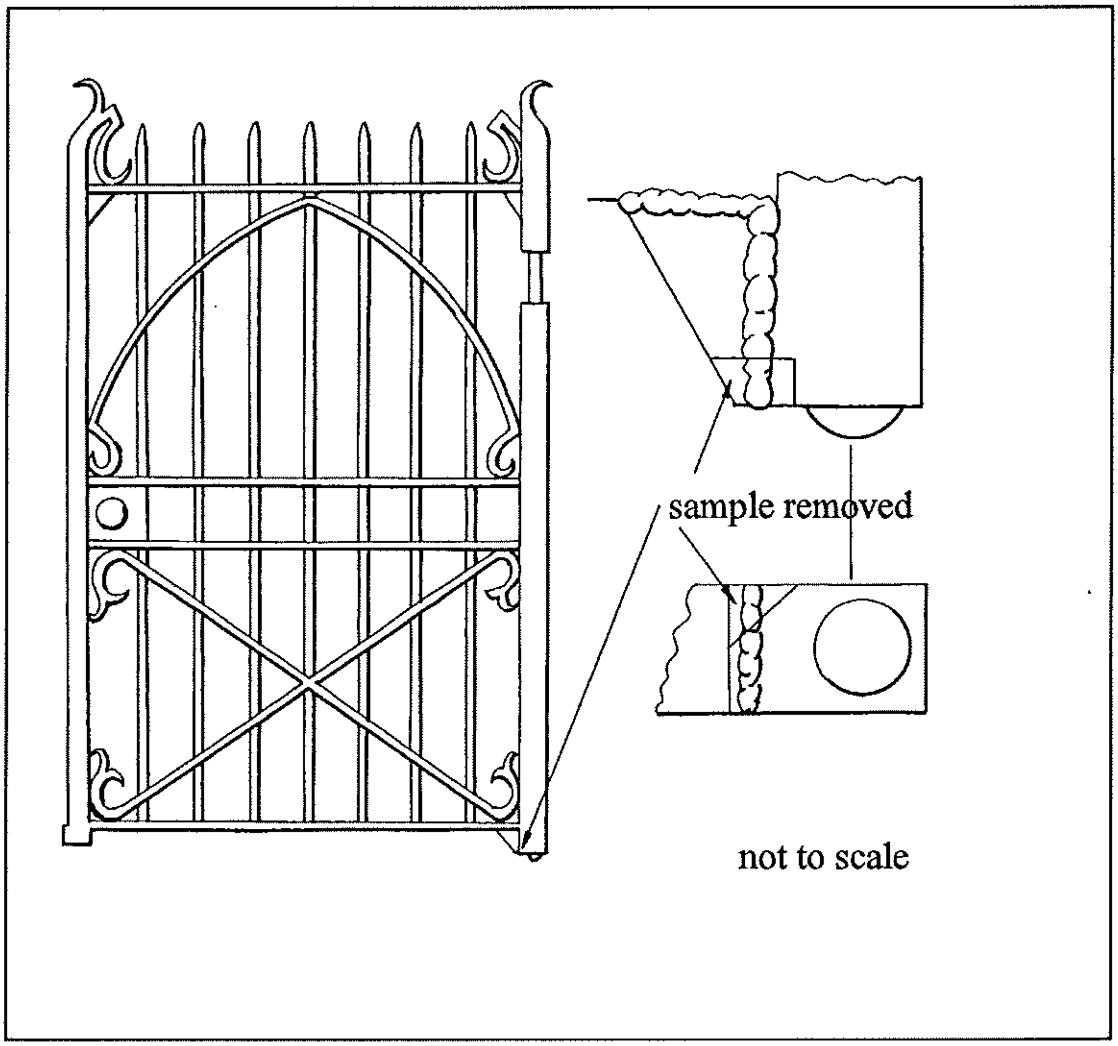

Figure 4 Garrison church gate

Metallographic Preparation

Samples were removed as indicated in Figures 3 and 4. A complete square section from the bottom of a vertical rail was removed. This was then cut in half so that the structure could be examined in longitudinal and transverse section. An angled cut across the bottom of the gate upright gave a sample which showed the upright in longitudinal section together with a portion of the reinforcing fillet and the weld metal that joined the two. Both samples were mounted in conductive phenolic resin and prepared using standard metallographic techniques; grinding on successively finer abrasive papers then polishing with diamond impregnated cloths. The specimen was examined on a metallurgical microscope in both the "as polished" i.e., unetched condition and after etching in 2% nital (nitric acid in alcohol). A Shimadzu microhardness tester was used to determine the hardness of different phases within the metallographic structure.

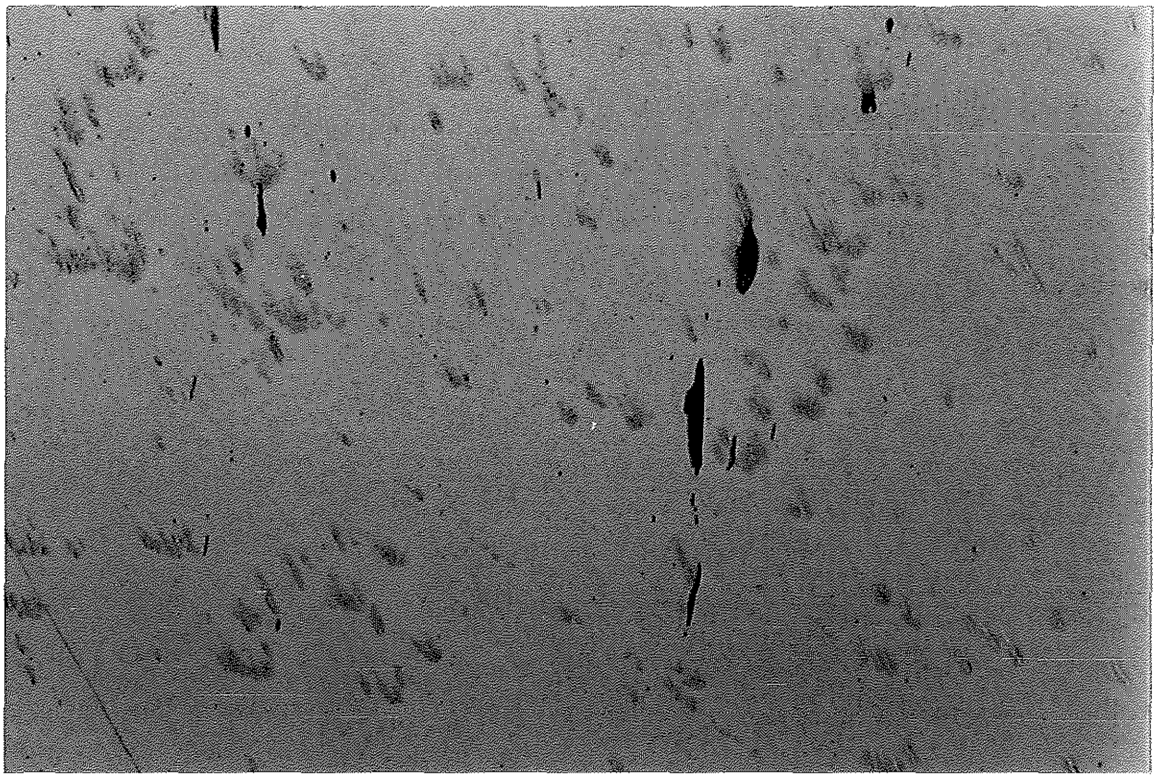

Plate 5 Garrison Church railing. Micrograph: unetched x400
Slag inclusion elongated along length of bar

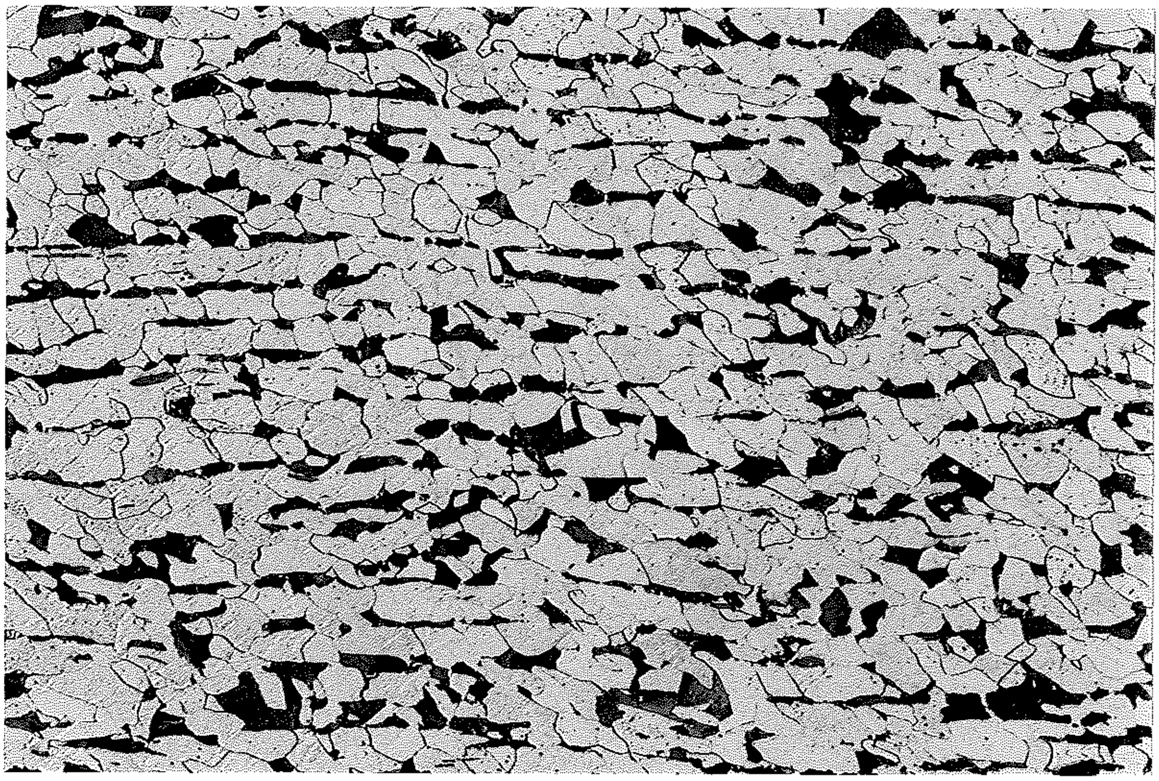

Plate 6 Garrison Church railing. Micrograph: etched in 2% nital x200
Banded ferrite (75%) and pearlite 25%

Metallographic Structure

1) Wrought iron railing

Viewing in the **unetched** condition (Plate 5) single phase slag inclusions, occupying less than 1% of the surface of the sample, were seen to be elongated along the length of the bar. **Etching** in 2% nital (Plate 6) revealed a relatively high carbon content with slight banding of ferritic iron and pearlite. Overall the elongated grains of ferrite, of size ASTM 6 comprised 75% of the structure, with the remaining 24% being fine pearlite.

Microhardness tests on the metal phases of the rail gave the following values (50g load, 10sec. load time):

ferrite	184.8 H_v	ferrite	191.6H_v
longitudinal section	178.4 H_v	transverse section	191.6 H_v
	227.0 H_v		204.4 H_v
	167.9 H_v		191.6 H_v
	210.0 H_v		195.1 H_v
mean	193.6 H_v		194.8 H_v

2) Gate

At the location of the sample it could be seen that the weld metal was not physically joined to the gate upright, a film of slag separated the two. **Unetched** (Plate 7) the upright was found to contain about 1% slag stringers, elongated along the bar, and sometimes containing two discernable phases. The fillet contained similar, if slightly fewer slag inclusions, whereas those in the weld metal were of spheroidal morphology. **Etching** in 2% Nital showed that, away from the weld zone, both the fillet and the upright were of very similar structure to the rail (Plate 8), although the grain size was slightly larger (ASTM 5 and 6 respectively), perhaps resulting from slightly higher rolling temperatures. The weld metal had a columnar, Widmanstätten structure radiating from the interface with the fillet (Plate 9). Ferrite was present as grain boundary allotriomorphs, widmanstätten side plates and intragranular plates. The remaining structure being of upper bainite. In the proximity of the weld the metallic structure of the fillet had been altered to form three distinct bands (Plate 10). Closest to the weld, the grain coarsened heat affected zone was of Widmanstätten form (ASTM 4) with ferrite at the grain boundaries and martensite/bainite within. Further from the weld the grain refined heat affected zone contained Widmanstätten of ASTM 7 with grain boundary ferrite and pearlite in the centre. Beyond this zone the grain size was unaltered, but the pearlite had spheroidised.

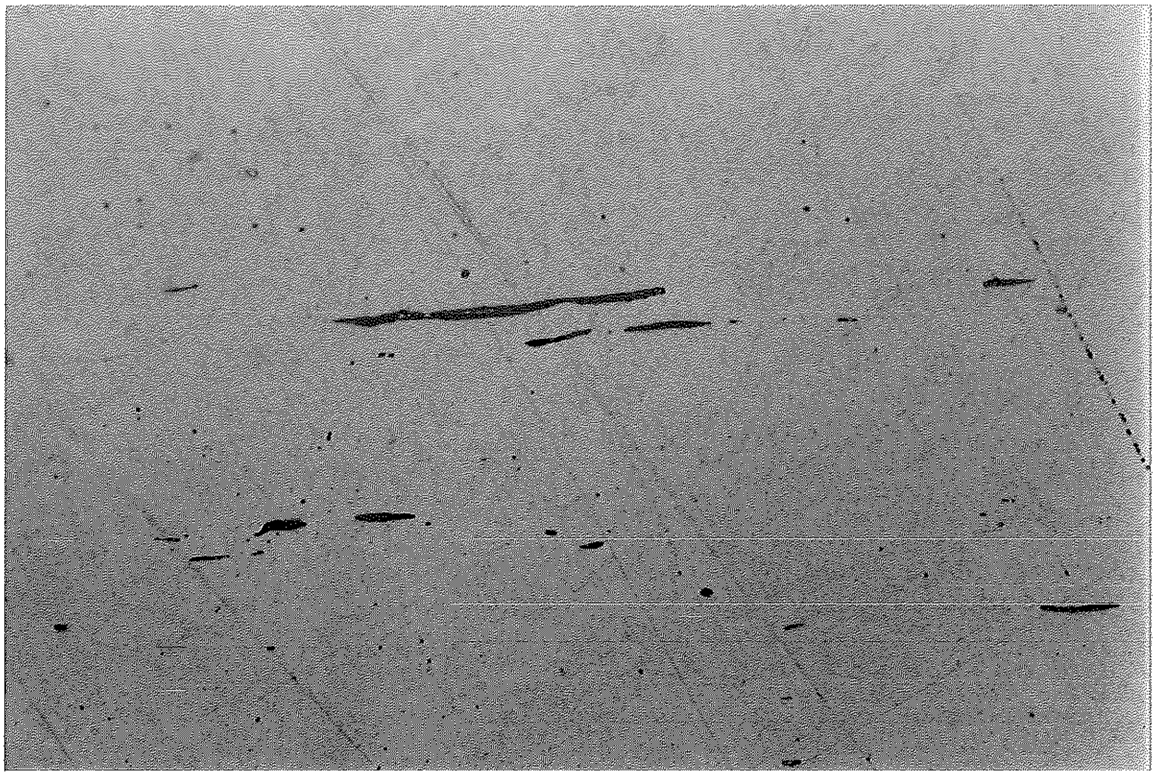

Plate 7 Garrison Church gate. Micrograph: unetched x400
Slag stringers elongated along length of bar

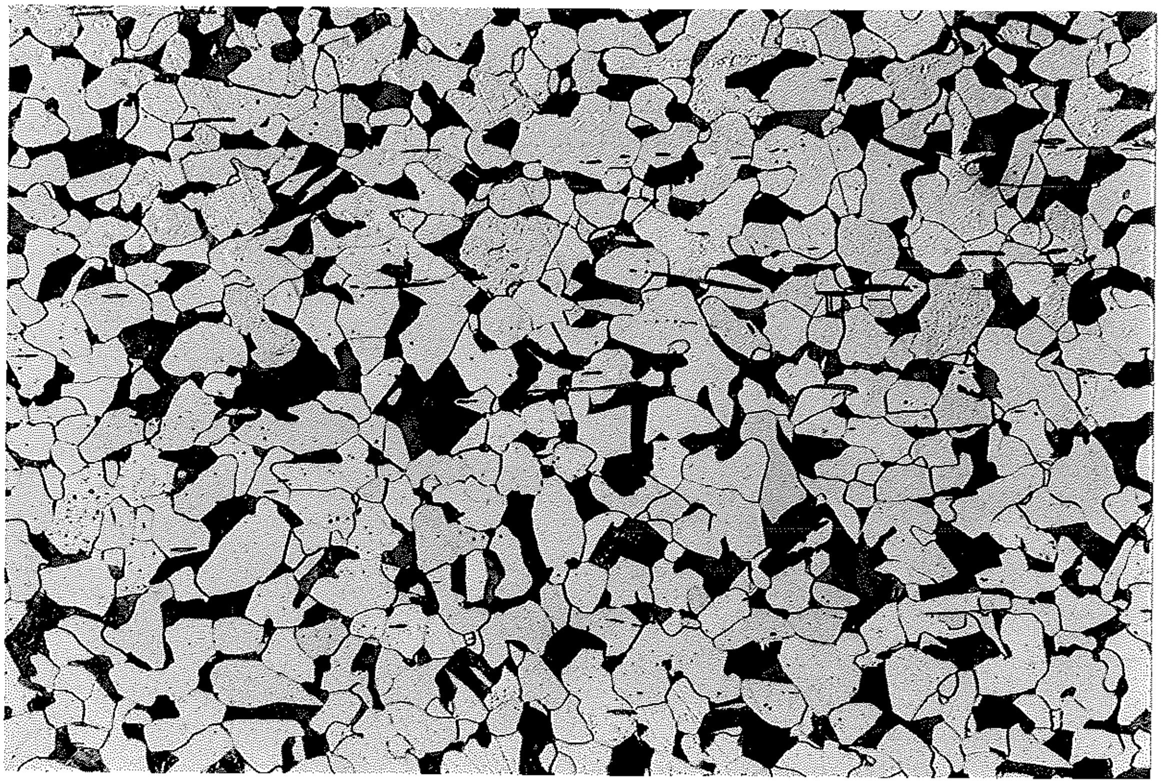

Plate 8 Garrison Church gate. Micrograph: etched in 2% nital x200
Ferrite and pearlite

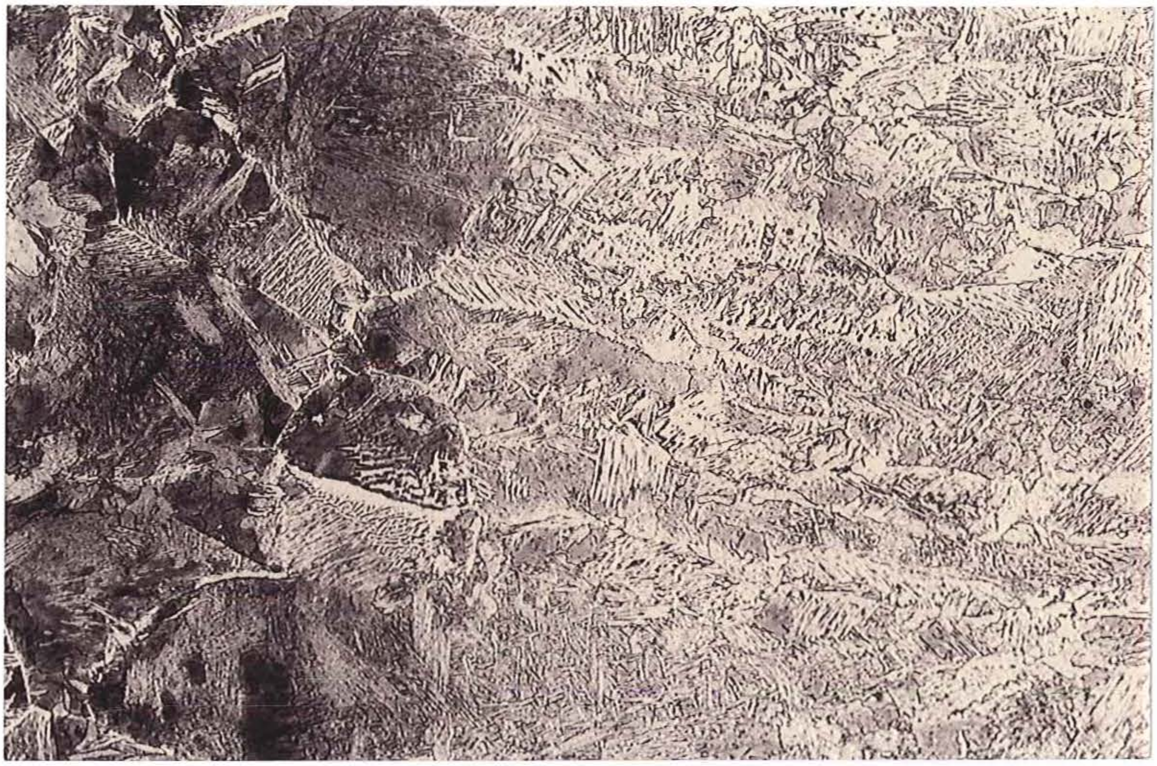

Plate 9 Garrison Church gate. Micrograph etched x200
Left: heat affected zone to left, Right: weld metal with widmanstätten structure

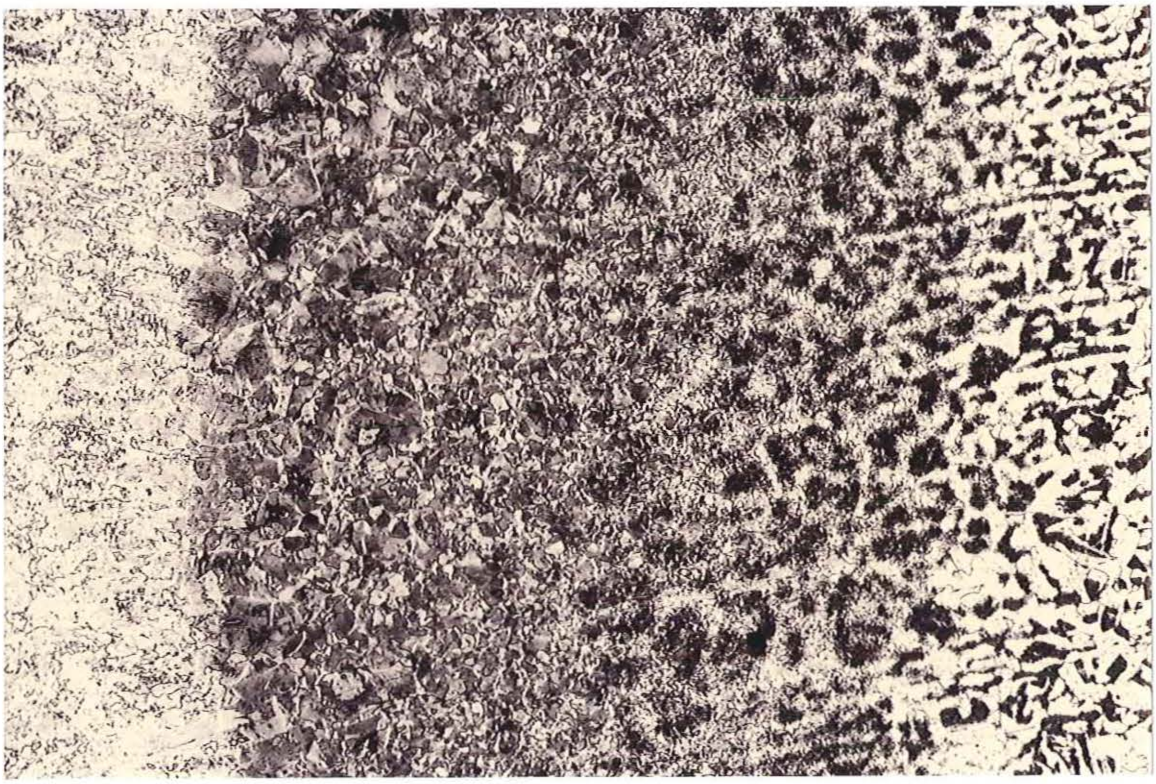

Plate 10 Garrison Church gate. Micrograph: etched in 2% nital x200
Left: weld metal, right: fillet

Microhardness tests on the metal phases of the gate gave the following values (50g load, 10sec. load time):

upright ferrite	125.3 H_v	fillet ferrite	126.3 H_v	weld metal	204.4 H_v
	141.5 H_v		113.4 H_v	bainite	270.7 H_v
	158.3 H_v		133.0 H_v		276.9 H_v
	139.3 H_v		127.2 H_v		279.9 H_v
	134.0 H_v		117.4 H_v		270.9 H_v
mean	139.7 H_v		123.5 H_v		260.6 H_v

Interpretation of the Structure and discussion

The original fabric of the railing and gate, which includes the triangular strengthening fillets, is 'wrought iron', containing a significant carbon content, approximately 0.2%. The dating of such material is not easy as a number of processes were used in the past to produce what is now termed 'wrought iron'. The traditional bloomery process was the only smelting technique used in Britain until the introduction of the blast furnace in 1496. However, the relatively small bloom produced in a bloomery would not have shown such homogeneous carbon content as that found in the railing and gate.

The product of the blast furnace; cast iron, must undergo further processing to produce a malleable 'wrought iron' suitable for forging. Originally this was undertaken in a charcoal fired finery, but from the late eighteenth century puddled iron was produced in reverberatory furnaces which allowed coal to be used as fuel. In both these techniques the low melting point cast iron was remelted in oxidizing conditions which, as it reduced the carbon content also raised the melting range until a pasty mass of iron containing a significant quantity of silicate slags resulted. Most commonly, the process was continued until almost no carbon remained, however it was possible to terminate the process earlier to retain enough carbon for steel to be produced. This partial decarburisation was evidently not easily controlled, but its advantages, which included the removal of sulphur and phosphorus from the iron could not be matched by the early 'acid' Bessemer process.

The Bessemer converter, introduced in the late 1850s, provided a cheap alternative to wrought iron, in which the high temperatures achieved allowed slag to float free of the liquid iron or steel. Difficulties encountered by Bessemer in removing phosphorus were overcome by the basic oxygen process, but puddled iron continued to be favoured for architectural wrought iron features, to the extent that in the early 20th century a process was developed in the United States to add synthetic slag to Bessemer iron. In Britain the last puddling furnace closed as recently as 1972.

Thus it is not easy to date wrought iron or determine its technological origins. In this example the only clue would appear to be the very uniform carbon distribution, which would tend to suggest the puddling process, the large scale of which allowed homogeneous alloys to be produced. The significant carbon content of the iron, bordering on a low carbon steel, is unusual. Although it would have imparted greater toughness to the metal, this would not have been a great advantage for such, largely ornamental, features.

Metallographic examination of the weld metal and its relatively narrow heat affected zone indicates that this results from arc welding rather than oxy-acetylene. Early patents for arc welding date from 1849, but the technique only became widely practicable with the introduction of consumable bare steel rods in 1888. As mentioned above, the lack of any attempt to clean up the welded area, suggests a somewhat makeshift later attempt to repair the gate rather than its original manufacture. It is, therefore not possible to state whether the gate, and railings were constructed before, during of after the 1868 restoration of the church. It is, however clear that the repairs to the gate post date the restoration by at least 20 years.