

# *Forging and Corrosion Resistance of the Delhi Iron Pillar*

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## **Abstract**

The effect of manufacturing methodology on the corrosion resistance the Delhi iron pillar has been addressed. The distribution of entrapped slag particles, surface stress state, surface finish and local surface compositions have been discussed. The presence of elongated slags in the microstructure and compressive stresses on the surface are beneficial for the adherence of the protective passive film. The benefits of enrichment of P that occurs on the surface due to the relatively high temperatures employed to soak and then forge-weld the phosphoric iron have been described. The significant contributions of the manufacturing technology, imparting favorable material structures and compositions in the surface regions, in enhancing the corrosion resistance of the Delhi iron pillar have been highlighted.

## **Introduction**

The iron pillar (Figure 1) located in the courtyard of the Quwwat-ul-Islam mosque, adjacent to the Qutub Minar, in New Delhi is world famous for its exceptional resistance to atmospheric corrosion. Moreover, the pillar's exquisite artistic construction continues to marvel present-day visitors. Metallurgists, corrosion scientists and archaeologists have evinced great interest in the pillar, keen on unraveling the hidden mysteries of the pillar<sup>1,2</sup>. The pillar was built during the reign of Chandragupta II Vikramaditya (375-413 AD) of the Imperial Gupta dynasty and it was originally installed in front of a Vishnu temple in Udayagiri in Central India<sup>3</sup>. It was moved to its current location in Delhi sometime in the 13<sup>th</sup> Century AD by Iltutmish<sup>4</sup>.

The intimate relationship between processing methodology and the ensuing material properties is well known in materials engineering. Additionally, the structure and performance of the engineering product is also related to these two factors. It is, therefore, important to explore the effect of the forging methodology employed to construct the Delhi iron pillar on its excellent atmospheric corrosion resistance. The present communication will address and discuss the effect of forging methodology on the corrosion resistance of the Delhi iron pillar.

## **Corrosion Resistance**

The pillar obtains its excellent corrosion resistance due to the formation of a protective passive film on the surface. The constituents of the protective passive film have been identified by modern state-of-art characterization methods<sup>5</sup>. The protective passive film consists of crystalline iron hydrogen phosphate hydrate

( $\text{FePO}_4 \cdot \text{H}_3\text{PO}_4 \cdot 4\text{H}_2\text{O}$ ), goethite ( $\alpha\text{FeOOH}$ ), lepidocrocite ( $\gamma\text{FeOOH}$ ), misawite (amorphous  $\delta\text{FeOOH}$ ) and magnetite ( $\text{Fe}_{3-x}\text{O}_4$ ). The iron oxide/oxyhydroxides were present in the amorphous form. The process of protective rust formation on Delhi pillar iron has been explained based on the rust analysis<sup>6</sup>. The pillar iron contains a relatively high percentage of phosphorus, the reason for which would be described later. Initially, the corrosion rate of iron is high due to the presence of entrapped slag inclusions. This results in enhancement of surface P content. The formation of a protective compact layer of misawite (amorphous  $\delta\text{FeOOH}$ ) is catalyzed next to the metal surface in the presence of P and this confers the initial corrosion resistance. The critical factor aiding the superior corrosion resistance of the Delhi iron pillar, however, is the formation of iron hydrogen phosphate hydrate, next to the metal-metal oxide interface. The transformation of the amorphous phosphate to the crystalline state is aided by alternate wetting and drying cycles (i.e. the environmental factor). The rate of corrosion is further lowered due to the lower porosity content of the crystalline phosphate phase. Additionally, repeated wetting and dry cycling results in conversion of the oxyhydroxides to nanosized and amorphous structures, as indicated by the characterization results<sup>5</sup>. This would also reduce the porosity in the outer layers of the atmospheric rust, thereby further contributing positively to the corrosion resistance. The relatively moderate climate of Delhi and the large mass of the pillar are also contributing factors that cannot be fully discounted. It is important to emphasize that the Delhi pillar iron exhibits excellent corrosion resistance only on atmospheric exposure. If the Delhi pillar iron is subjected to corrosion under immersed conditions (i.e. soil, aqueous electrolyte, etc.), it would perform similar to ordinary irons. This is attested by the severe corrosion of the pillar in the buried underground region<sup>2</sup>.

### **Manufacturing Methodology**

The co-relation between processing, structures, properties and performance is well established in materials engineering. Therefore, the effect of manufacturing methodology will have a significant bearing on the pillar's corrosion resistance. The Delhi iron pillar was manufactured by the forge-welding technique. The most likely manufacturing methodology, based on critical analyses of various aspects like hammering method, heating method, forging method, use of inserts, use of dies and ease of handling, has been described in detail elsewhere<sup>2,7</sup>. It is briefly described to provide an overview of the process (Figure 2). The starting material for construction of the pillar were individual iron lumps, obtained from the iron extraction process. Ancient Indians did not possess the knowledge of melting iron because the ancient iron-making furnaces (called bloomery furnaces) could not attain the high temperatures required for melting iron. Iron was produced by solid state reduction of iron ore with the aid of charcoal (Figure 2). As limestone was not used in the charge, the entrapped slag inclusions found in ancient irons are essentially fayalitic and the

CaO contents in the slags are insignificant. This resulted in a higher P content in ancient irons. The presence of higher P has a beneficial effect on the atmospheric corrosion of iron. The solid-state reduced bloom, from the bloomery furnace, was hammered immediately after removal from the furnace in order to remove the still-liquid slags present in the pores of the bloom. As it was impossible to remove the slag completely, some of these were invariably retained in the pancake on cooling down the hammered bloom. This is the origin of entrapped slag inclusions in ancient Indian irons, in general. Iron alloys obtained by the solid-state reduction route are called wrought irons.

As all the iron pancakes were not produced at the same time in the same furnace, it is important to realize that the compositions and microstructures of the individual pancakes would not be precisely the same. However, the iron pancakes were of fairly consistent quality as attested by the known compositions of the Delhi pillar iron (Table 1). The material of the pillar is not homogeneous. This inherent inhomogeneity in the material is not unique to the Delhi pillar iron, but is, in fact, a common feature of ancient Indian irons that have been extracted by the bloomery process.

The manufacturing methodology would be briefly reviewed. The heated iron pancakes were placed on the side surface of the pillar and hammered on to the same by the use of hand-held hammers (Fig. 2). The addition of metal was sideways with the pillar positioned in the horizontal direction. Therefore, the process can be termed as horizontal forge welding. Visual and scientific proofs for this manufacturing methodology have been described in detail elsewhere<sup>7</sup>. The entrapped slag inclusions in the iron are elongated<sup>12</sup> along the vertical axis of the pillar, thereby indicating that the dynamic force was applied in a direction perpendicular to the vertical axis in order to forge weld the pancake on to the surface. The pillar's vertical and horizontal movements were aided by handling clamps and rotating pegs provided on the surface of the pillar (Fig. 2), the protruding portions of which were chiseled off during the surface finishing operations. Finally, the surface of the pillar (that was supposed to be exposed above the ground) was smoothed by surface hammering, chiseling and burnishing, thereby providing it a relatively smooth and tapered cylindrical appearance. The surface that was to be originally buried under the ground was not finished to a smooth surface. It was rather left in a rough surface condition to aid better gripping of the pillar with the base surrounding. A part of the original buried surface is still visible in the lower region of the Delhi iron pillar because these portions were exposed when the pillar was re-erected in the Quwwat-ul-Islam mosque. Lastly, the Sanskrit inscription was inscribed on the surface of the pillar using cold dies. The decorative bell capital was finally fit on to the top portion of the main body and then the whole pillar erected in the main courtyard of the Vishnu temple.

The description of the manufacturing methodology of the pillar has been

provided in order to elucidate its effects on the pillar's corrosion resistance. The Delhi iron pillar is an excellent example of ancient India's forge welding technology. Hadfield made the following comments after carefully weighing his words, "the Delhi iron pillar was of a better quality than anything they were able to produce today."<sup>8</sup>

## Discussion

The elongation of the entrapped slag inclusions, the state of stress in the surface regions and the compositional variations that result as a result of the forge welding operation will be analyzed in order to elucidate the effect of manufacturing methodology on the corrosion resistance. The application of surface coatings during manufacture would be first addressed as some earlier investigators have discussed this possibility as a part of the pillar's manufacturing methodology. The application of intentional surface coatings to provide corrosion resistance has now been discounted based on modern observations<sup>2</sup>.

### *Surface Coatings*

Herrero and Zubiria<sup>13</sup> attributed the pillar's excellent corrosion resistance to a kind of fine slag coating on the surface, which they speculated to originate from the manufacturing process. They hypothesized that the removal of the slag coating would cause the pillar to corrode severely.

A slightly different modified scheme of protective film, formed during manufacture, was proposed by Bardgett and Stanners<sup>12</sup>. They reasoned that the continued hammering process to produce the smooth surface of the pillar in the final stages of manufacture must have been performed with the iron in the hot condition and that the process must have taken considerable time. They suggested that an oxide film, along with slag inclusions, could have been hammered on to the surface during this operation. The presence of slag inclusions in the surface oxide was proposed to enhance the protective ability of the scale. Additionally, Bardgett and Stanners maintained that the pillar would have taken a long time to cool down owing to its large heat capacity and this would have allowed the surface oxide scale to further develop. They hypothesized that the quality of the oxide produced by the sequence of manufacturing operations promoted the preservation of the pillar in the relatively dry atmospheric climate.

It has also been proposed<sup>14</sup> that some special surface treatments were provided to the pillar during manufacture, like Bower-Barff process. Bower-Barff coating is the name for the protective coating developed on iron, that was in vogue in the early part of the last century. In this process, red hot iron was treated in steam at high temperatures, which lead to the formation of magnetite (black oxide)  $\text{Fe}_3\text{O}_4$  without the formation of the intermediate sesquioxide  $\text{Fe}_2\text{O}_3$  (red rust) by the reaction  $3\text{Fe} + 4\text{H}_2\text{O} \rightarrow \text{Fe}_3\text{O}_4 + 4\text{H}_2$ . Wood<sup>14</sup>, based on the appearance of the pillar surface, ascribed the excellent corrosion resistance of the pillar to it having been subjected to a process

similar to the Bower-Barff process immediately after manufacture. This suggestion was reiterated by Rosenhain<sup>15</sup> without providing any proof.

It has also been speculated that regular application of oil would have resulted in a protective layer when the pillar was originally located in the Hindu temple. However, this practice must have been discontinued when the pillar was relocated in the mosque. Moreover, the top portion of the pillar would not have been applied with oil as it was inaccessible to the public and this region also shows no significant signs of rusting. In this regard, Evans<sup>16</sup> mentioned that the effect of handling with perspiring fingers leads to corrosion rather than corrosion resistance because salt and lactic acid are applied to the surface by this method.

Finally, in tune with the recent scientific interest in nanomaterials, it has also been speculated that the iron pillar may have been coated with nano powders of iron alloyed with phosphorus<sup>17</sup>.

The above theories of artificial surface coatings are not valid because it has been explained that the protective passive film forms from within the material<sup>6</sup>. A visual proof of the same is that a freshly cut surface of the pillar attains the color of the rest of the pillar in approximately a short duration of time (about three years)<sup>18</sup>.

### ***Slag Envelopes***

Lahiri *et al*<sup>10</sup> analyzed the microstructure of Delhi pillar iron and observed that it contained high fraction of slag inclusions dispersed in three dimensions. The microstructures revealed elongated slag inclusions near the forge welded zones along with severe mechanical distortions. They reasoned that, after the metal blocks were forge welded, the entire pillar was rounded off through application of local forge hammering resulting in surface cold working and elongation of slag inclusions. They suggested that the slag inclusions created three dimensional slag envelopes around the metal grains and this resulted in corrosion resistance. Therefore, they attributed the corrosion resistance of the Delhi pillar to the mode of manufacture and fabrication, which resulted in a three-dimensional envelope of slag around metal grains. However, this view cannot be supported based on the available microstructures of the Delhi pillar iron<sup>9,19</sup>. They also quoted the slag coating proposal of Herrero and Zubiria<sup>13</sup> while arriving at their conclusion.

The interfaces between individual iron pancakes were also flattened in the direction along the vertical axis of the pillar. Therefore, any corrosion front will have to encounter these interfaces before it could have proceeded further. The presence of elongated slag stringers and the lump-lump interfaces aid in stopping the corrosion front from moving forward. This idea has also been proposed by Lahiri *et al*<sup>10</sup> to be one of the causes for the pillar's corrosion resistance. However, this may be an overstatement because the pillar is never really in immersed conditions and it is exposed only to much milder atmospheric environment exposure. Therefore, severe

corrosion is not anticipated and there is no real need to invoke the presence of these elongated slag stringers in preventing the progress of the corrosion front. It is also important at this juncture to point out that the dispersion of the slag particles would be important when pitting attack initiates and progresses in the interfacial region between the entrapped slag inclusions and the matrix. Pitting attack would apply when immersed conditions, like burial in soil conditions, are involved and in such cases, the role of slag inclusions becomes important. The slag inclusions provide a physical pathway for solutions, containing dangerous ions like chloride, to penetrate deep into the metal and establish corrosion cells. The removal of these ions is almost impossible from the crevices along the slag inclusions. However, if the slags are not interconnected, by sufficiently breaking them up and thus isolating them, then high slag inclusion densities should not have any effect on localized corrosion. The slag inclusions in the Delhi pillar iron are dispersed and not interconnected<sup>10,12</sup>, which is one of the important consequences of the forging operations. Therefore, the dispersed slag inclusions are not harmful in allowing localized attack to progress deep into the material.

### ***Entrapped Slag Elongation***

The entrapped slag inclusions are elongated in a direction perpendicular to the direction of force application due to the application of dynamic force during the forge welding operation. In the case of the Delhi iron pillar, the slag particles are elongated in the vertical axis direction<sup>12</sup>. If an iron pancake that has been forge welded on the pillar is hypothetically analyzed, the structure of the internal entrapped slag inclusions would be as shown schematically in Figure 3. The elongated slag inclusions in the HP (hammering plane) would offer protection against movement of the corrosion front, whereas the long transverse (LT) and short transverse (ST) cross-sections are prone to severe localized corrosion due to the nature of dispersion of the elongated slag inclusions. A similar relationship has been experimentally verified for wrought iron by Chilton and Evans<sup>20</sup>. The most severe corrosion was found in the LT and ST faces, located within narrow bands. On the other hand, the HP exhibited maximum resistance because the movement of the advancing corrosion front was hindered by the presence of delineations of slag inclusions. The nature of corrosion attack in three directions in a Gupta-period iron clamp from the Eran temple has also been studied<sup>21</sup>. Severe localized corrosion, in the form of localized bands, resulted due to the relatively large fraction of entrapped slag inclusions that were observed in the LT and ST sections<sup>21</sup>. The attack was severe in the ST and LT faces because the higher density of slag-metal interfaces in these sections compared to the hammering plane<sup>21</sup>.

In the case of the Delhi iron pillar, the surface exposed to the atmosphere is the HP (depicted in Figure 3) and therefore, the most corrosion resistant. The other two faces of the forged pancake (i.e. ST and LT) are never exposed to the environment because of the cylindrical shape of the pillar. Therefore, it can be firmly concluded

that the sideway forging of iron pancakes on the pillar by the horizontal forge welding technology contributed positively to the corrosion resistance of the pillar. In the process of manufacturing the Delhi pillar, the slag inclusions have been elongated such that the hammering plane was exposed to the environment. Therefore, the method of manufacture ensured that the most corrosion resistant face, after forging operation, was exposed to the atmosphere. This is a beneficial effect of forge welding on corrosion resistance, wherein the forging methodology resulted in a favorable alignment of the entrapped slag inclusions, which offer excellent resistance against progress of the corrosion front. Had the pillar been forge welded vertically (i.e. with the pillar in the vertical position and the addition of pancakes to the radial cross section of the pillar) as has been hypothesized by some authors<sup>1,22</sup>, then the LT and ST sections of the forge-welded lumps would have been exposed to the environment. This would have greatly aggravated the corrosion of the pillar. It could probably be for this important reason that the pillar was not manufactured by the vertical forge welding technology.

It must be realized that pitting, caused by the presence of entrapped slag inclusions, will not necessarily be a problem for the HP section exposed to the environment. If the atmospheric corrosion initiates pitting faster than protective film formation, then localized corrosion of the surface would be a major problem. On the other hand, if the protective passive film covers the surface faster than the initiation of pitting, then there would be no pitting of the surface. The latter situation appears to apply in the case of Delhi iron pillar, with respect to the entrapped slag inclusions in the iron. However, some of the interfaces between individual iron lumps, that are exposed to the environment, exhibit signs of preferential attack, the cause of which (i.e. presence of lead fillings) has been described in detail elsewhere<sup>23</sup>.

The entrapped slags also exert a beneficial effect in the mechanical sense because they help in good adherence of the protective passive film to the surface. This has been found in the case of wrought iron where the surface film were generally more adherent than on mild steel<sup>20</sup>. Apart from these, the role of slag inclusions in aiding passive film formation, by enhancing the cathodic reactions on the surface, has been explained in detail elsewhere<sup>24</sup>.

### ***Stress State***

As the lumps were added to the pillar by forge welding, the surface regions are in a state of compressive stress. The distribution of residual stresses is important in determining corrosion attack. The propagation of corrosive attack is limited in the presence of compressive stresses and therefore, Evans first proposed that the compressive state of stress on the surface may have contributed positively to the pillar's corrosion resistance<sup>17</sup>. He reasoned that it would be difficult for corrosion to propagate through the compressively stressed surface region, and that hammering of the surface would have reduced the probability for initiation of attack<sup>17</sup>. If localized

corrosion is delayed by the presence of surface compressive stresses, the protective film can cover the surface faster than the initiation of localized attack. Another beneficial effect of the compressive stress state is that it aids the adherence of the surface protective passive film.

### ***Surface Finish***

It has been found that phosphate coatings are effectively adherent on a sand blasted surface<sup>25</sup>. Sand blasting created both compressive stresses as well as a relatively rough surface. The sand blasted surface possesses many active centers and sites for nucleation, aiding the precipitation of passive films. The slag inclusions can be considered as active centers for passive film nucleation. However, very rough sandblasted surfaces are not ideal as the nucleation of the phosphates is not uniform and yields an irregular passive layer. The Delhi pillar in the exposed regions, exhibits a relatively smooth surface finish. This smooth finish, which is a consequence of the final manufacturing operation, aids protective passive film adherence.

### ***Surface Decarburization***

Relatively, the carbon content in the Delhi iron pillar is low while the P content is high (Table 1). The non-uniform heterogeneous composition is another characteristic of ancient Indian irons. The heterogeneities in composition and microstructures in the Delhi pillar iron and their effect on protective film formation have been discussed in detail elsewhere<sup>19</sup>. Special attention would be provided to the surface regions in the discussion below. One of the characteristics of ancient Indian irons is that the surface regions of the objects are generally ferritic in nature with hardly any carbon is present in the near-surface regions<sup>2,9,21</sup>.

A sample was cut from the Delhi iron pillar in 1959 by researchers from the National Metallurgical Laboratory and the microstructures of the iron were observed at several locations within the material, starting from the surface<sup>9</sup>. Some of the microstructures are provided in Figure 4. The surface regions are almost depleted of carbon and the structure is almost pure ferrite. Coarse grains of ferrite were observed near the surface (Figure 4a, b and c) and they generally possessed slip bands (see Fig. 4b). The surface was free from pearlite. Small to medium grains of ferrite were seen in the next area along with a small amount of pearlite at the grain boundaries. The amount of pearlite increased on progressing towards the interior<sup>9</sup>. Therefore, the microstructural results clearly establish the decarburized nature of the surface layers. The interesting aspect of surface decarburization is that P will segregate into regions where carbon is deficient<sup>26</sup> and therefore, the surface regions will be enriched in P during the decarburization process. Moreover, P is a ferrite stabilizer<sup>27</sup> and the presence of ferritic structure in the near surface regions is not surprising. A higher surface content of P is beneficial to the atmospheric corrosion resistance because of the formation of a protective passive phosphate film on the surface<sup>6</sup>.

A brief digression will address the effect of P on mechanical behaviour and this will help in understanding the deformation processing of Delhi pillar iron. In modern iron and steel making operations, the element phosphorus is maintained at levels lower than 0.05% because it is considered harmful. The presence of P leads to embrittlement of iron during cold working and also to enhanced temper embrittlement<sup>27</sup>. It is now known that the harmful effects of P is realized only when carbon is also present in the alloy in significant amounts<sup>27</sup>. The mechanical behaviour of Fe-P alloys reveals interesting features<sup>27</sup>. The presence of a very small amount of carbon actually offsets the embrittling effects of P. Therefore, in archaeological iron, the presence of high P along with low carbon contents was beneficial to the mechanical properties. The widespread use of phosphoric irons in ancient times can thus be appreciated. However, in modern technology, high P containing irons are deliberately avoided.

The temperature of soaking and forge welding are important in the manufacturing process and needs to be understood. As the P contents are higher in the Delhi iron pillar, the processing of the pillar must have been performed in the hot working range when ferrite is relatively soft and, further, stabilized by P. It has been experimentally verified that forging of iron-phosphorus alloys is relatively easy around 900°C<sup>27</sup>. Although the temperature of forge welding of the Delhi pillar is not known, it can be assumed that the forging must have been performed in the hot condition rather than cold. While undergoing the high temperature soak and during actual forging operation, conditions ideal for decarburization are achieved in the pancakes. Therefore, the origin for the decarburized structure observed in the near surface regions of the Delhi pillar iron must be the high temperatures that the iron pancakes were subjected to, when they were placed in the reheating furnace before forge welding and during the operation. The high heat capacity of the pillar would have further aided the surface decarburization (and the consequent surface phosphorous enrichment) of the Delhi pillar iron, because longer cooling times are required for the high heat capacity pillar. Therefore, the high temperature soaking and hot working operations are also beneficial to the corrosion resistance of the pillar because they aid the enrichment of P in the near-surface regions.

## **Conclusions**

The relationship between forging methodology employed to manufacture the Delhi iron pillar and its corrosion resistance has been described. The forging methodology affects the distribution of entrapped slag particles, surface stress state, surface finish and local surface compositions. It is concluded that the presence of elongated slags in the microstructure and compressive stresses on the surface are beneficial for the adherence of the protective passive film, thereby improving the corrosion resistance. Moreover, the enrichment of P that occurs on the surface, due to the relatively high temperatures employed to soak and then forge-weld the phosphoric iron, is directly

beneficial to the atmospheric corrosion resistance of the pillar. The significant contributions of the manufacturing technology in enhancing the corrosion resistance of the Delhi iron pillar have been highlighted. The horizontal forge welding methodology was directly beneficial in imparting material structures and compositions in the surface regions that were ably suited for imparting superior atmospheric corrosion resistance to the Delhi iron pillar.

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**TABLE 1**

<b>Published Composition Analyses of Delhi Pillar Iron</b>					
	Hadfield <sup>8</sup>	Ghosh <sup>9</sup>		Lahiri <i>et al</i> <sup>10</sup>	Lal <sup>11</sup>
	(1912)	(1963) Above	(1963) Under	(1963)	(1945)
C	0.08	0.23	0.03	0.26	0.90
Si	0.046	0.026	0.004	0.056	0.048
S	0.006	trace	0.008	0.003	0.007
P	0.114	0.280*	0.436-0.48	0.155	0.174
Mn	Nil	Nil	Nil	Mil	Nil
N	-	0.0065			
Fe	99.720	Diff			99.67
Others	0.246				0.011
Specific gravity	7.81	7.672-7.747		7.5	

\* 0.100 fixed and 0.180 in solid solution P

\* \* \*



Figure 1: The corrosion resistant iron pillar situated in the Quwwat-ul-Islam mosque in New Delhi.

## List of Figures

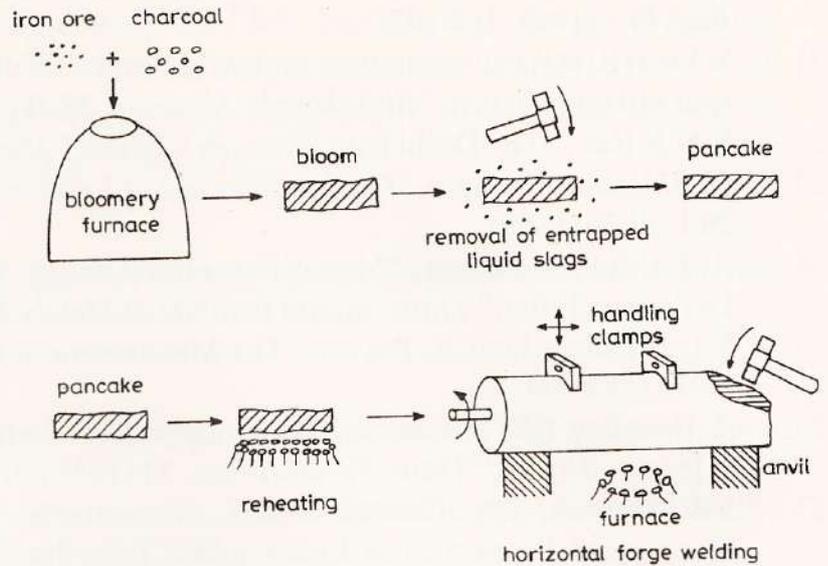


Figure 2: Schematic of the manufacturing methodology of the Delhi iron pillar (from iron making to horizontal forge welding).

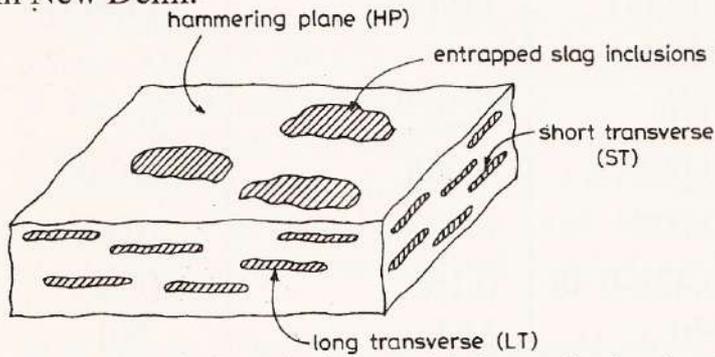


Figure 3: Schematic of the forge welded pancake showing the distribution of the elongated slag inclusions on hammering plane (HP), long transverse (LT) and short transverse (ST) sections.

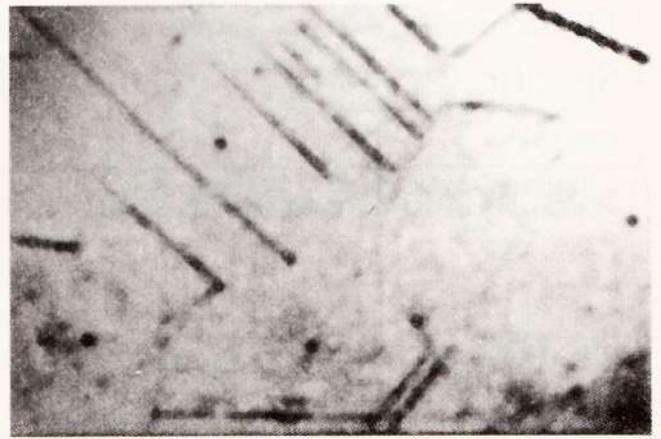
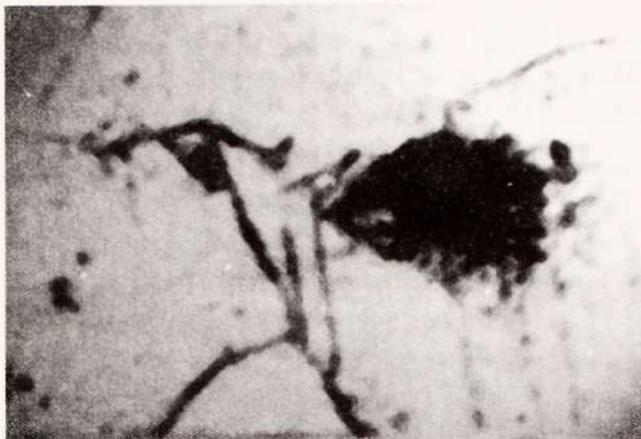


Figure 4: Microstructure of Delhi iron pillar from the near-surface regions<sup>9</sup>. (a), (b) and (c) Notice the ferritic structure at the surface with coarse grain sizes. Slip bands can be observed in (b) indicating deformation processing.