Abstract

The microstructure of the main structural elements (Phoenix columns, transversal beams and tie-rods) of the Simón Bolivar iron bridge, located in Arequipa (Peru), was investigated. The bridge was supposedly designed by Gustave Eiffel and opened to the public in 1882. The characterisation revealed that the columns, beams and tie-rods are made of puddled iron (ferritic matrix of equiaxial grains and numerous slag inclusions). The tensile properties were estimated by hardness and nanohardness measurements, and the tensile strength varied between 285 and 390 MPa and the Young's modulus from 187 to 198 GPa. The components of puddled iron with the same provenance, Phoenix Iron Company, showed typical variations in the chemical composition and microstructure of the slag inclusions. The microanalysis results of the slag inclusions indicated that the wüstite phase presented qualitatively the same composition with pronounced variations in the ratio between V$_2$O$_5$ and TiO$_2$ contents, confirming that a single parameter cannot be used to determine the provenance of iron artefacts. The intense presence of slag inclusions in the puddled iron components (~ 10% in volume fraction) heterogeneously distributed in the iron illustrates the technological limits of the iron refining technique of the time.

Keywords: puddled iron; microstructural characterisation; Simón Bolívar bridge; archaeological heritage of Peru.
1. Introduction

The Simón Bolívar bridge, also known as El Puente de Hierro, is one of the cultural heritage sites of Aréquipa, Peru, being declared as a Historical Monument of Peru in 1986. This bridge has a length of 488 m and was inaugurated in 1882 as part of the railway circuit that connected the coastal area to Aréquipa, towards Puno, Cuzco and then Tacna. The construction belongs to a railway bridge typology that uses diagonal trusses and risers, Phoenix columns and Fink trusses (Gutiérrez-Pinto, 2013). The Phoenix column, patented by Samuel Reeves in 1862, is a hollow cylinder made up of four, six, or eight wrought iron segments riveted together. This column is lighter and stronger than the cast-iron columns – round bars - used at the time, allowing the construction of bridges that support more mechanical loading. All of the bridge structural elements were produced by the Phoenix Iron Company, Philadelphia, USA, (Hillstrom K., 2005). This project presents typical lightweight characteristics of late 19th century American bridges, but many still credit this project to the French engineer Gustave Eiffel (Gutiérrez-Pinto, 2013). Some of these symbolic iron structures from the 19th century are withstanding the ageing process and have become part of the cultural heritage. One of the main challenges to maintain and safely extend the service life of these historical metallic structures lies in the understanding of the manufacturing processes available at that time (Scheremans et al., 2018). The grade and quality of the structural iron produced in the 19th-century are critical parameters for the repair and maintenance of the operational bridges (Holowaty and Wichowski, 2016). In this sense, the microstructure and mechanical properties of the structural components of iron railway bridges are critical for the cultural heritage conservation plans (Mayorga et al., 2015; Kowal and Szala, 2020).

Stötzel et al. (Stötzel et al., 1997) used fracture mechanics to determine the residual safety and service life of old steel bridges produced from 1850 to 1930 (in old riveted bridges, wrought and puddled iron were identified). The wrought iron presents similar chemical composition and microstructure to low strength-low alloy steels so they could use fracture mechanics to assess the safety of the bridges. Puddled irons, however, presented a different microstructure (lamellar morphology containing from ferrite and large slag inclusions) and chemical composition (oxygen content above 0.5% and manganese content below 0.1%). In the American state of Indiana, there are bridges with wrought iron truss structures, which have been in service since the late 19th century. Most of these bridges need some degree of maintenance or rehabilitation, and an investigation was carried out to review and classify the typical properties of wrought iron components. Wrought iron mainly consists of a ferritic matrix with numerous slag inclusions, leading to lower values of tensile strength when compared to most commercial steels. The average tensile strength of the wrought iron was 324 MPa, while the tensile strength was 378 MPa. From numerous results, rehabilitation and repair recommendations for the wrought iron components were developed (Bowman and Piskorowski, 2004). Mayorga et al. (Mayorga et al., 2015) investigated the fatigue mechanism of parts of the 1862 puddled iron railway bridge and showed that the large slag inclusions were related to the brittle behaviour of the iron, while the smaller slag inclusion was related to the microplasticity. According to Revilla (Revilla, 2005), there are numerous structures built with puddled iron during the 19th century, which remain in use today. Therefore, structural integrity assessments are required to extend the life and establish surveillance and maintenance programs of these structures. The author presented a fracture mechanics methodology for evaluating the structural integrity of old riveted beams using puddled iron samples from two Spanish bridges built in the late 19th century. The microstructure, chemical composition, hardness, tensile properties and fracture toughness of the puddled iron were investigated. The elastic modulus varied from 120 to 190 GPa, the yield strength from 200 to 245 MPa, the tensile strength from 245 to 390 MPa, the elongation from 1.4 (transversal direction) to 15% (longitudinal direction), indicating the heterogeneity and anisotropy of tensile properties caused by the microstructure of the puddled iron. Kowal and Szala (Kowal and Szala, M., 2020) diagnosed the microstructure and mechanical properties of a puddled iron railway bridges built between 1894 and 1898 in Poland. Fatigue, fracture, hardness and tensile tests were performed along with chemical composition and microstructural characterisation. The mechanical properties of the puddled iron components parts were compatible with the structural steel used for the bridge structures.

Since its inauguration, the Simón Bolívar bridge has gone from rail to vehicular and currently pedestrian-only usage. Throughout its 137 years of service, studies of the bridge’s structural integrity have never been performed to verify any damage caused by corrosion of the environment, as there is agricultural production under the bridge. This information will be critical to the bridge’s restoration and conservation plans. The objective of the present research is to characterise the chemical composition and microstructure of the structural elements of the bridge to estimate the tensile properties and identify the main features of the steelmaking process of the components.

2. Materials and methods

Samples were taken from different areas of the bridge, carefully removed with the permission of the Ministry of Culture and Management of the Historic Centre of Aréquipa. Small fragments of the Phoenix columns, transversal beams, tie-rods and the bases of the columns of the bridge were collected, representing the main structural ele-
ments of the bridge (see Figures 1a and 1b). Optical emission spectroscopy (OES) was used for the determination of the chemical composition of the puddled iron samples, following ASTM A415-15 standard. The metallographic samples were prepared using SiC abrasive grinding papers (from 500 to 1200 grits) and diamond polishing pastes (from 6 to 1μm). The samples were etched with 5% Nital for 5s and examined using optical and scanning electron microscopes, the latter equipped with x-ray energy dispersive spectroscopy spectrometry (EDS microanalysis) for the chemical microanalysis of the non-metallic inclusions. The volume fraction of the slag inclusions was analysed using "Image J" image analysis software. During the EDS microanalysis, the authors imposed that all the elements were in the form of oxides. For each sample, at least ten slag inclusions were EDS chemically analysed.

Hardness determination used the Rockwell B method (1/16" penetrator, 100kg load, Indectec durometer), according to ASTM E-18 standard, a minimum of 5 measurements were made for each sample. The Vickers microhardness measurements were carried out in a Shimadzu microhardness testing machine (300g load and 15 seconds) – a minimum of 10 microindentations for each sample.

The tensile strength values of the puddled iron components were estimated using an empirical equation obtained for the wrought iron samples of the Bell Ford Bridge (Bowman and Piskorowski, 2004). According to these authors, the tensile strength (MPa) can be roughly estimated by multiplying the hardness value (from Rockwell B Hardness Test) by 4.51. The average Young modulus was determined by the nanohardness method (4 measurements), which records the stress and displacement during loading and unloading (10mN load and 15 seconds) (Ma et al., 2004).

![Figure 1 - General view of the Simón Bolívar bridge. (a) Railway bridge is part of the Ferrocarilles del Sur, connecting the Peruvian south-pacific coast to Cusco and Puno (Gutierrez-Pinto, 2013); (b) Overview of the bridge base, indicating the Phoenix columns (arrow a), the transversal beams (arrow b), the tie-rods (arrow c) and the support of the Phoenix columns (arrow d).](image)

### 3. Results

The chemical analysis results of the wrought iron components are shown in Table 1. These results indicate that the carbon contents of the beam and columns are below 0.056% and that the phosphorus content is high, exceeding the maximum detection limit of the method, which is 0.15%. The sulphur content varied from 0.07 to 0.15%, and the high silicon (between 1.52 and 2.98%) and phosphorus (above 0.15%) contents are probably due to the of these elements in the slag inclusions (fayalite matrix). The aluminium content is also very high for all the components, while the nickel content varies from 0.02 to 2.01%; chromium content ranges from 0.01 to 0.15%; vanadium content varies from 0.03 to 0.17%, and cobalt content varies from 0.02 to 0.22%, showing the typical variation in the chemical composition of puddled irons in the XIX century, which nowadays would characterise different classes of materials. According to the American Society for Testing Materials (ASTM), the puddled iron is a refined material containing an "evenly" distributed amount of slag. Puddled iron has "little chemical composition heterogeneity", and its average carbon content is between 0.018% and 0.030% (Oliveira, 2017; Bouw et al., 2009). According to Revilla (Revilla, 2005), the puddled irons made in Europe between 1850 and 1930 feature silicon levels close to 0.20%, while phosphorus levels reach 0.47%.

| Component                  | C    | Si  | P   | S   | Ni  | Cr  | Al  | V   | Co  | W   | Fe  |
|----------------------------|------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Beam (TP-1)                | 0.056| 1.52| >0.15| 0.12| 0.018| 0.15| 0.43| 0.17| 0.22| 0.37| 96.1|
| Phoenix column (PP-2)      | 0.035| 2.98| >0.15| >0.15| 2.01| 0.029| 0.32| 0.046| 0.03| 0.20| 93.3|
| Phoenix column (PP-5)      | 0.041| 1.16| >0.15| 0.072| 0.016| 0.013| 0.50| 0.026| 0.016| 0.20| 97.1|
The microstructures of the beams, tie-rods and columns show equiaxial ferrite grains, with grain size varying from 10 µm to 50 µm, and slag inclusions, see Figures 2a to 2d. The numerous and heterogeneous slag inclusions of different sizes occupy a volume fraction between 8 to 13%. Some of the small and globular slag inclusions pin the grain boundaries of the ferrite, while large multiphasic inclusions are elongated along the plastic deformation direction. The results of chemical composition and microscopy indicated that the beams, tie-rods and columns are made of puddled iron (Stötzel et al., 1997; Revilla, 2005). The large proportion of slag inclusions means that the puddling process was used to fabricate these wrought iron components instead of the modern liquid steelmaking technologies available in the second half of the XIX century. Puddled iron is a malleable material produced between the eighteenth and late nineteenth centuries from the decarburisation of the pig iron bath at temperatures slightly below the liquidus temperature. An iron spear would be introduced into this semi-solid pig iron mass and, when rotated, this spear would aggregate iron and slag until forming a 250 kg bloom. The bloom would be removed from the oven, following forging and rolling (Smith, 1882; Rostoker and Dvorak, 1990).

The slag inclusions of the puddled iron components were examined in more detail (see Figures 3a to 3d), and they are usually formed by a matrix of fayalite (Fe$_2$SiO$_4$, dark area, with the additional presence of Al$_2$O$_3$. P$_2$O$_5$), and dendrites of wüstite (FeO, light area). Additionally, the slag inclusions of the tie-rod samples presented islands of metallic Fe (small white regions) and hercynite precipitation (Al$_2$O$_3$. FeO, black areas), see Figures 3c and 3d (Okafor, 1992; Photos, 1987; Gordon, 1997). The components of puddled irons with the same provenance (Phoenix Iron Company) showed variations in the chemical composition and microstructure of the slag inclusions. The EDS chemical microanalysis results of the wüstite phase of the slag inclusions are shown in Table 2. The chemical composition of the slag inclusion can be used to investigate the provenance of ferrous artefacts (Dillmann and L’Héritier, 2007; Blakelock et al., 2009; Maia et al., 2015; Mamani-Calcina et al., 2017). The microanalysis results indicated that the wüstite phase of the beams, tie-rods and columns qualitatively presented the same chemical composition, with pronounced variations in the ratio between V$_2$O$_5$ and TiO$_2$ contents.

Rockwell B hardness results (see Table 3) were used to estimate the tensile strength of the ferrite phase of the columns and beams made of puddled iron, using an equation obtained for the wrought iron samples of the Bell Ford Bridge (Bowman and Piskorowski, 2004). The Vickers microhardness of the ferritic matrix of the puddled irons (see Table 4) showed values between 135 and 185HV, which are compatible with the variations in the chemical compositions and possible presence of inclusions underneath the indentation marks (see Table 1). The results of Young’s modules for the ferrite of the puddled iron obtained by nanoindentation showed average values of 195 GPa (experimental values of 198 GPa, transversal beam, sample 4 TP-1; 198 GPa, Phoenix column, sample 6 PP2; 192 GPa, transversal beam, sample 7 TP-3, and 187 GPa, Phoenix column, sample 13 PP-5). Finally, the microstructure of the grey cast iron, found in the bases of the Phoenix columns, revealed type A graphite flakes in a ferritic-pearlitic matrix and presence of steadite, a ternary eutectic microconstituent commonly found in pig iron, see Figure 4 (Stefanescu, 2008; Hillert and Söderholm, 1975).

Figure 2 - Microstructural characterisation of the puddled irons, showing multiphasic slag inclusions in pure Fe matrix. (a) Transversal beam, sample 4, TP-1; (b) Phoenix column, sample 13, PP-5; (c) Tie-rod, sample M2; (d) Tie-rod, sample M4. Optical microscopy. Etching: 5% Nital.
Figure 3 - Microstructural detail of the slag inclusions showing dendrites of wüstite (FeO, light grey area) surrounded by fayalite (Fe₂SiO₄, dark grey area). (a) Transversal beam, sample 17, TP-7; (b) Phoenix column, sample 13, PP-5; (c) and (d) Tie-rod, sample M4. Additional metallic Fe islands (small white regions), hercynite precipitation (Al₂O₃·FeO, black regions) and oxi-sulphide precipitation (small light grey areas, see Figure d). Scanning electron microscopy, backscattered electron image.

Table 2 - Chemical microanalysis of the wüstite phase of the slag inclusions of the puddled iron of the beam, tie-rod and column (average of 10 measurements for each sample).

| Sample                  | Oxide   | (%)   | Sample                  | Oxide   | (%)   | Sample                  | Oxide   | (%)   |
|-------------------------|---------|-------|-------------------------|---------|-------|-------------------------|---------|-------|
| Transversal beam        | Al₂O₃   | 0.2   | Tie-rod (sample 207 M4) | Al₂O₃   | 0.5   | Phoenix column          | Al₂O₃   | 0.3   |
| (sample 4, TP-1)        | SiO₂    | 0.3   |                         | SiO₂    | 1.2   |                         | SiO₂    | 0.3   |
|                         | TiO₂    | 0.4   |                         | TiO₂    | 0.8   |                         | TiO₂    | 0.4   |
|                         | V₂O₅    | 1.0   |                         | V₂O₅    | 0.4   |                         | V₂O₅    | 0.6   |
|                         | MnO     | -     |                         | MnO     | -     |                         | MnO     | 0.2   |
|                         | FeO     | 97.9  |                         | FeO     | 97.2  |                         | FeO     | 98.3  |

Table 3 - Rockwell B hardness measurements and the estimated tensile strength (average of 5 measurements for each sample).

| Sample | Hardness (HRB) | Tensile strength (MPa) | Sample | Hardness (HRB) | Tensile strength (MPa) |
|--------|----------------|------------------------|--------|----------------|------------------------|
| Beam   | 87             | 392                    | Column | 75             | 339                    |
| Beam   | 63             | 284                    | Column | 84             | 379                    |
| Beam   | 81             | 366                    | Column | 71             | 320                    |
| Beam   | 74             | 334                    | Column | 73             | 330                    |
| Beam   | 70             | 316                    | Column | 73             | 330                    |
| Beam   | 63             | 285                    | Column | 70             | 316                    |
| Average| 73.0           | 329                    | Average| 74.3           | 335                    |
Table 4 - Vickers microhardness values of the ferrite phase in the puddled iron samples (average of 10 measurements for each sample).

| Sample | Hardness (HV) | Sample | Hardness (HV) | Sample | Hardness (HV) |
|--------|--------------|--------|--------------|--------|--------------|
| Beam   | 139          | Column | 135          | Tie-rod| 140          |
| Beam   | 153          | Column | 142          | Tie-rod| 132          |
| Beam   | 151          | Column | 160          | Tie-rod| 177          |
| Beam   | 145          | Column | 143          | Tie-rod| 185          |
| Average| 147          | Average| 145          | Average| 160          |

Figure 4 - (a) and (b) Microstructure of the support of the Phoenix column. Ferritic-pearlitic grey cast iron with the presence of steadite (rod-like eutectic, white) and inclusions (light grey). Optical microscopy. Etching: 5% Nital.

4. Discussion

Puddled irons can be identified by their amount of slag inclusions and heterogeneous distribution of ferrite grains (Störzel et al., 1997; Revilla, 2005). The puddled iron samples of the bridge (Phoenix columns, tie-rods and transverse beams) featured a maximum carbon content of 0.056% and high values of silicon (between 1.52 and 2.98%), phosphorus (above 0.15%) and sulphur (from 0.07 to 0.15%) (see Table 1). The high phosphorus content of the puddled irons is in agreement with the values found in other historical ironwork structures, such as 0.47% of P (Revilla, 2005) and 0.25 to 0.36% of P (Bowman and Piskorowski, 2004). The low microhardness values of the ferrite (see Table 4) reinforce the idea that silicon and phosphorus contents in the ferrite are low due to their presence in the slag inclusions, which was confirmed by the presence of P and Si in the fayalite matrix.

The microstructures of the puddled irons revealed a matrix of equiaxial ferritic grains with varying sizes and a significant amount of multiphasic slag inclusions (volumetric fraction from 8 to 13%), see Figures 2a to 2d. The presence of slag inclusions in the iron samples differentiates the puddled iron from much cleaner Bessemer and Siemens-Martin steels, which were beginning to be used at that time (Revilla, 2005). The slag inclusions of the puddled irons presented a duplex microstructure, see Figures 3a and 3b, consisting of a matrix of fayalite (Fe₂SiO₄) with the additional presence of Al₂O₃, P₂O₅ and dendrites of wüstite (FeO), see Table 2. Additionally, the slag inclusions of the tie-rod samples presented islands of metallic Fe, and precipitation of oxi-sulphides and hercynite, see Figures 3c and 3d (Okafor, 1992; Photos, 1987; Gordon, 1997).

The puddled irons with the same provenance (The Phoenix Iron Company) showed typical variations in the chemical composition (see Table 1) and microstructure of the slag inclusions (see Figures 2a to 3d). The EDS chemical microanalysis results (see Table 2) showed that the wüstite phase of the beams, tie-rods and columns presented qualitatively the same chemical composition, with distinct variations in the ratio between V₂O₅ and TiO₂ contents, indicating that a single parameter cannot be used to determine the provenance of iron artefacts, endorsing previous results (Dillmann and L’Héririer, 2007; Blakelock et al., 2009; Mamani-Calcina et al., 2015; Mamani-Calcina et al., 2017).

The microanalysis results of the slag inclusions of the puddled iron produced by an American manufacturer, Phoenix Iron Company, see Table 2, were compared to the results of the puddled iron of the structural elements of the Dom Pedro II Bridge - located in Bahia, 1885, and produced by a Scottish company, Mossend Iron and Steel Works (Mamani-Calcina et al., 2017). The wüstite phase of the slag inclusions of the American puddled iron presented higher V₂O₅ (0.8%) and lower TiO₂ (0.45 %) and MnO (0.1%) contents than the Scottish puddled iron (0.5% V₂O₅, 0.9% TiO₂ and 0.9% MnO). Additionally, the Scottish puddled iron featured a lower level of inclusion content (approximately 6%) than the American puddled iron (about 10%). These results illustrate how puddled irons of different provenances feature slag inclusions with different content and chemical composition (Dillmann and L’Héririer, 2007; Blakelock et al., 2009; Maia et al., 2015; Mamani-Calcina et al., 2017).

The estimated tensile strength values for the structural elements made of puddled iron (Phoenix columns and transversal beams) are between 285 and 390 MPa, see Table 3. These values are...
higher than the values usually found for modern commercially pure iron in the annealed condition, which are between 204 and 212 GPa (CES Edupack, 2019). However, these values are compatible with the values found for puddled iron in the literature, such as the average tensile strength of 378 MPa (Stötzel et al., 1997) and the experimental tensile strength interval ranging from 245 to 390 MPa (Revilla, 2005). The assessment of the tensile strength, however, disregarded the presence of "hard and brittle" slag inclusions (volumetric fraction of approximately 10%). In this sense, the values of the tensile strength of the puddled iron structural elements should be lower than the estimated values (Table 3), due to the reduction of the effective resistant area by the presence of slag inclusions, and the action of these inclusions as stress concentrators. For instance, the stress concentration factor of a plate with a circular hole, where the ratio between the diameter of the hole and the remaining width is equal to 0.1, is around 2.5 (Boresi and Schmidt, 2003).

The average value of the Young's modulus of the puddled iron, 194 GPa, is slightly below the typical values found for commercially pure iron in the annealed condition, which are between 204 and 212 GPa (CES Edupack, 2019). This value, however, is compatible with the values presented for puddled iron in the literature, such as the experimental interval ranging from 120 to 190 MPa, depending on the orientation of the ferrite-inclusion "lamellar" microstructure to the tensile stress (Revilla, 2005). Finally, the bases of the Phoenix columns and tie-rods were manufactured with ferritic-pearlitic grey cast iron, which nowadays features typical tensile strength values between 65 and 100 MPa (CES Edupack, 2019).

5. Conclusions

1. Analysis of the results of microstructure, volume fraction of slag inclusions and chemical composition, indicates that the transverse beams, tie-rods and Phoenix columns of the Simón Bolivar Bridge were manufactured from puddled iron, while the bases of the Phoenix columns were made of ferritic-pearlitic grey cast iron.

2. Puddled irons with the same provenance (Phoenix Iron Company) show typical variations in the microstructure of the slag inclusions and bulk chemical composition.

3. The slag inclusions were formed by a matrix of fayalite and dendrites of wüstite, with the occasional presence of islands of metallic Fe, and precipitates of oxi-sulphides and hercynite.

4. The microanalysis results of the slag inclusions indicated that the wüstite phase presented the same chemical composition with pronounced variations in the ratio between V_2O_5 and TiO_2 contents, confirming that a single parameter cannot be used to determine the provenance of iron artefacts.

5. The wustite phase of the slag inclusions of the American puddled iron (Phoenix Iron Company) presented higher V_2O_5 and lower TiO_2 and MnO contents than the Scottish puddled iron (Mossend Iron and Steel Works). The Scottish puddled iron featured a lower level of inclusion content (approximately 6%) than the American puddled iron (about 10%). These findings illustrate how puddled irons of different provenances feature different slag inclusions.

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