

Revealing the iron skeleton of Notre-Dame de Paris. Methodologies and issues of archaeological and analytical investigations following the blaze

Maxime L'Héritier, Aurélia Azéma, Delphine Syvilay, Delphine Neff, Ivan Guillot, Emmanuelle Delqué-Kolic, Thibault Creacheadec, Philippe Dillmann

► To cite this version:

Maxime L'Héritier, Aurélia Azéma, Delphine Syvilay, Delphine Neff, Ivan Guillot, et al.. Revealing the iron skeleton of Notre-Dame de Paris. Methodologies and issues of archaeological and analytical investigations following the blaze. Journal of Cultural Heritage, 2024, 65, pp.78-86. 10.1016/j.culher.2023.05.006. hal-04103863

HAL Id: hal-04103863 https://hal.science/hal-04103863

Submitted on 2 Jan 2024

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution - NonCommercial - NoDerivatives 4.0 International License

L'Héritier M., Azéma A., Syvilay D., Neff D., Guillot I., Delqué-Kolic E., Creacheadec T., Dillmann P. Revealing the iron skeleton of Notre-Dame de Paris. Methodologies and issues of archaeological and analytical investigations following the blaze.

Abstract

The fire at Notre-Dame de Paris led to the discovery of several series of previously unknown iron armatures, which were systematically recorded. Among them, several iron staples from the top of the eaves walls, lead crest reinforcements and a sample of the tie rods used in the choir in the 19th century were analysed by metallography and tensile tests in order to determine their nature and mechanical characteristics. These results can be put into perspective with previous studies on such monuments. The chemical analysis with SEM-EDS of the slag inclusions contained in the matrix of these ferrous alloys makes it possible to determine the technical processes used for their production throughout the construction and restoration of the building. This information is complemented by a series of radiocarbon dates currently being carried out to better understand the periods of use of these ferrous reinforcements. When compared with those of other materials, these dates could be used to recalibrate the phasing of the building. Finally, the development of a methodological tool combining microscopic study of the corrosion layers and structural analysis (µ-Raman) and based on comparisons with a reference corpus from Metz cathedral, has made it possible to determine the temperatures reached by the staples at the top of the walls in order to better understand the dynamics of the fire and its degradation.

Keywords: Iron armatures, Gothic construction, Dating, Metallography, Tensile tests

1. Introduction

Iron armatures were commonly used in the masonry of many medieval monuments, especially for the construction of Gothic churches. Archaeological observations performed over the last decades have proven that since the beginnings of the 13th century, many great cathedrals implemented iron cramps, chains or tie-rods in their initial designs in order to reinforce the masonry during construction and sometimes as means of consolidations [1,2]. This is notably the case for Chartres and Bourges cathedral, which are considered so far as the first gothic churches with a systematic use of iron armatures (tie-rods and chains for Bourges and series of cramps for Chartres) [3–5]. A few decades later the cathedrals of Beauvais, Coutances or Troyes, for example, or the abbey church of Saint-Denis and the Sainte-Chapelle seem to have perfectly integrated the use of this material in monumental stone architecture [6–11]. Several tons of iron are then used to reinforce masonries, but also in the windows to maintain stained glass panels as well as to attach numerous ornamental elements, such as pinnacles or statues.

The same picture was not observed so far for 12th century gothic cathedrals, such as Laon or Noyon were the use of iron seems to be limited to a few pins. Notre-Dame de Paris belongs to the same impulse of Gothic churches construction in the middle of the 12th century, being the latest and greatest of these first Gothic cathedrals. So far, the use of iron in its structure has not been documented [12], apart from Viollet-le-Duc's comments in his Dictionnary of Architecture [13]. During the 19th century restorations, he noticed the presence of several rows of cramps surrounding the choir of the building, which he dates from 1195 without further comments.

« La grande corniche à damiers qui couronne le chœur de la cathédrale de Paris, et qui dut être posée vers 1195, se compose de trois assises de pierre dure formant parpaing, dont les morceaux sont tous réunis ensemble par deux rangs de crampons, ainsi que l'indique la [figure 1.a]. Cela constituait, au sommet de l'édifice, au-dessus des voûtes, un puissant chaînage... » (tome 2, article "chaînage")¹

Moreover, the records of the work of the Historical Documents Commission for the year 1846 report that Lassus adopted a new method of chaining of this part of Notre-Dame cathedral using large sections chains consolidating the two upper courses [14]. This system is still visible from the outside surrounding the top of the chevet. Despite their possible importance to the structure of the building, these 19th century elements were so far never studied (figure 1).



Figure 1 : (a) Rows of cramps in the chequered cornice of the choir as described by Viollet-le-Duc in his Dictonnary of Architecture. (b) Reinforcement of the same part of the building by iron chains in the 19th c. by Lassus.

The destruction brought by the 2019 fire has uncovered many other uses of metal in Notre-Dame's structure notably discovered in the burnt remnants of the upper parts. The temperatures of the fire were high enough to melt lead and sometimes copper alloys but not iron (melting at 1535 °C). Most of these armatures can be associated with the 19th century restorations according to their use (in the spire of for cresting for example) or shape (machined armatures). However, many forged elements, which might belong to earlier medieval or modern construction or restoration phases, were also discovered as well as other metallic structures, still in place yet invisible before the fire. The presence of these unknown and/or unstudied armatures has to be questioned in terms of archaeology but also in the scope of the building's restoration.

2. Research aims

The discovery of these iron armatures led to the development of an original research program, bringing together experts in the field of iron in the CNRS/MCC scientific project and taking into account the questions raised by the architects and the contracting authority (EPND). This paper aims to present the different uses of iron in Notre-Dame throughout the construction and restoration phases of the building and the historical and heritage issues associated with this material. How, in such a context of new discoveries and considering the scope of the restoration work that has begun, can we question these ancient iron armatures from a historical and archaeological perspective also focused on the

¹ Translation : "The large chequered cornice which crowns the choir of Paris Cathedral, and which must have been laid around 1195, is composed of three courses of large hard stone joining both sides of the wall, the pieces of which are all joined together by two rows of spikes, as shown in [figure 1.a]. This formed, at the top of the building, above the vaults, a powerful chain..."

conservation/restoration of this exceptional heritage? The mobilisation of the reflections and knowledge acquired over more than twenty years of research on ancient building irons [1] will be presented with a view to better understanding the construction of the cathedral, in the service of its restoration, together with some first results and the development of new methodological approaches specifically built around the case of Notre-Dame.

3. Materials & Methods: the use of iron in Notre-Dame de Paris

Extensive prospection was carried out in Notre-Dame following the 2019 fire to highlight the use of iron reinforcements in its masonry. Access to the upper parts of the building was possible thanks to harnessing and scaffolding. Some parts of the upper walls, located under the provisional roof in the choir and in the transepts were however not accessible. However, no geophysical means of detection could be performed in the upper masonries due to lack of access in the exterior of the building. Moreover, the burnt remnants of the framework and spire were excavated according to the protocol presented in this issue [15]. All iron artefacts (pins, nails and different kinds of armatures), were collected and sorted.

The result of these prospections and excavation shows that iron armatures were used at different levels in Notre-Dame's structure and correspond to different construction phases from the medieval parts of the building to the 19th century restorations and adjunctions (figure 2).

First, the builders used a great number of cramps in Notre-Dame's masonry as soon as the tribunes' level and up to the upper walls (figure 2 a & b). Viollet-le-Duc describes the presence of cramps in the chequered cornice of the choir yet other cramps were revealed on the top of the eaves-bearing walls, right below the burnt framework (figure 3). All these reinforcements (tribunes, cornice and upper walls) are made of two rows of 40 to 50 cm long cramps (60 to 70 including both legs), sometimes on several courses. Smaller cramps (25 cm) are also implemented to attach the monolithic columns blocks decorating the chapels' walls: each of the three 8 m high column circling each pier is made of two to three en delit blocks of stone, whose upper part is joined to the wall using such cramps (figure 2 c & d). The same feature is used for the piers situated in between the inner and outer aisles with blocks up to 4 m high and only about 16 cm in diameter. These series of cramps seem to have different functions: attaching fragile slender en delit columns to the masonry, reinforcing the upper walls maybe due to the framework thrusts and maybe providing a provisional buttressing to the great arcades in the outer aisles. A more extensive discussion on their role is presented elsewhere [16].

As in every gothic church, the builders also use iron bars and small rods to attach the stained glass panels and to reinforce the windows' structure. Today, most of the iron bars still in place dates from the 19th or 20th century following the loss of medieval stained glass and the great restauration campaigns. However, in the upper bays, which are nowadays glazed with transparent glass, the vestiges of bars are still visible at the top of the bay mullions (figure 2 e). They were probably cut during one of the many restauration campaigns when the stained glass were taken down.

Another use of iron is linked to masonry reinforcement. We already mentioned that in 1846 architect Jean-Baptiste Lassus adopted a new reinforcement method for the two upper layers of the choir using iron chains. These chains are made of an outer chain embedded in the walls of the choir and of the eastern bays of the transept at the height of the chequered cornice and an inner chain, located alongside the walls at the same level. Transverse armatures crossing the wall link both chains, and transverse tie-rods located above the vaults join the inner chains to one another. The fire affected the inner chains and tie-rods and some parts were slackened or even broken.

Many iron armatures were found in the excavation of the vestiges of the framework. Most are machined bolted rods that could have been integrated in the 18th or most probably 19th centuries works. Other pegs and nails were however hand-forged. A corpus of forged nails and brads (headless nails used for roofing) of different sizes (from about 1 cm long to more than 30 cm) was isolated for study (figure 4). Their typology and repartition in the vaults allowed to identify bigger nails used in the framework and smaller (10-15 cm long and 15-25 g heavy) nails used in its suspended wooden pathway. Lastly, many iron pieces were integrated in the 19th century construction and restauration works to sustain lead ornaments. They are particularly numerous at the top of the roof for the cresting made of cast lead elements attached on an iron armature shaped in the form of leaves (figure 2 f and figure 3). Built by Viollet-le-Duc in the 19th centuries, it was strongly restorated during different renovation phases in the 20th century: the choir and southern transepts by Fonquernie in the late 1970's and 1980's and the northern transept by Mouton in the end of the 2000's. Many decorations from the spire also use such iron armatures (figure 2 g).

Several questions arise regarding this use of ferrous armatures in Notre-Dame's structure. They deal with the historical knowledge of the monument and its construction, but also with issues linked to the building's conservation and restoration, regarding for example the diagnosis of pathologies or even the question of reuse. These different questions will be presented in the following sections as well as the associated methodologies. Several iron armatures were therefore sampled, sometimes on demand of the architects and/or the commissioner (table 1). Each corpus will be detailed in the appropriate section and some first results will be briefly discussed.



Figure 2 : Iron armatures in Notre-Dame de Paris. (a) Cramps discovered on the top of the upper walls (b) cramps in the tribunes (c) & (d) cramps attaching monolithic columns blocs to the piers in the nave (e) ancient iron bars remains in the upper bays (f) iron armature of lead cresting (g) iron armature of spire lead decoration



Figure 3: Situation of some armatures in the choir of Notre-Dame (series of cramps in yellow, cresting in blue, tie-rods in red).



Figure 4: Different types of forged nails found in the excavation of the vaults debris.

Sample	Туре	Location	Metallography	Tensile	Radiocarbon
				tests	Dating
NN1	cramp	Upper walls (nave north)	Х	Х	Х
NN9	cramp	Upper walls (nave north)	Х	Х	
NS7	cramp	Upper walls (nave south)	Х		
NS8	cramp	Upper walls (nave south)	Х		Х
NS106	cramp	Upper walls (nave south)	Х		Х
VN2	cramp	Upper walls (nave south)	Х		
VN4	cramp	Upper walls (nave south)	Х	Х	
CH1	cramp	Upper walls (nave south)	Х	Х	Х
TS01	cramp	Upper walls (southern	Х		
		transept)			
GUA01	cramp	Nave chapels	X		
TRIB01	cramp	Tribunes (choir)	Х		Х

TRIB02	cramp	Tribunes (choir)	Х		Х
TIR01	cramp	Upper choir	Х	Х	
CF VN4	cresting	Nave roof	Х	Х	
CF NDP 232	cresting	Nave / Northern transept roof	Х	Х	
CF CH4	cresting	Choir roof	Х	Х	
CF TS BV	cresting	Southern transept roof	Х	Х	
CH14 MCL 01	Small nail	Choir framework (pathway)	Х		
CH14 MCL 03	Small nail	Choir framework (pathway)	Х		Х
CH11 MCL 01	Small nail	Choir framework (pathway)	Х		
CH11 MCL 04	Small nail	Choir framework (pathway)	Х		
CH11 MCL 06	Small nail	Choir framework (pathway)	Х		
CH11 MCL 08	Small nail	Choir framework (pathway)	Х		
CH11 GCL 28	Big nail	Choir framework	Х		
CH11 GCL 34	Big nail	Choir framework	Х		
CH11 GCL 47	Big nail	Choir framework	Х		
CH11 GCL 43	Big nail	Choir framework	Х		
CH11 GCL 46	Big nail	Choir framework	Х		
VN9 MCL 01	Small nail	Nave framework (pathway)	Х		
VN9 MCL 02	Small nail	Nave framework (pathway)	Х		
VN9 GCL 17	Big nail	Nave framework	Х		Х
Total			31	9	8

Table 1 : List of studied samples and analysis performed

4. Results & discussion: questions and methodologies for heritage iron armatures.

4.1 Ferrous alloys quality and the question of reuse.

Quality is among the first parameters regarding any construction material. Unlike wood or stone whose quality are mainly intrinsic given the wood type or the geological formation, ancient ferrous alloys are highly heterogeneous materials, whose qualities rely on several operations in their production chains: choice of ore, reduction process, cleansing or refining, forging.... Indeed, before the second half of the 19th century and the introduction of Bessemer and Thomas refining processes, iron was partly of totally produced at a solid state (i.e. below the melting temperature of the metal). There is therefore no homogenization of the ferrous alloy composition (regarding carbon or phosphorus content), which mainly depends on the heterogeneous thermodynamic and kinetics conditions in the shaft furnace. Moreover, several second phase particles (slag inclusions) coming from the smelting or refining stages remain entrapped within the metallic matrix. All these factors are likely to affect the mechanical characteristics of the iron armatures eventually produced. Determining the nature and quality of these alloys gives clues about the choices of materials that the builders made during construction, restauration or upkeep of the building. It is also of primary importance for the present restauration work, especially knowing that the 2019 fire might have weakened certain structures.

To answer these questions, 31 iron armatures were sampled: 12 cramps, 4 cresting, 14 nails and 1 internal tie-rod piece. Details about the sampling is given in table 1. Metallographic examinations were carried out on all samples using Nital etching on all cross sections to reveal carburised zones [17], as well as Oberhoffer's reagent on a selection of sections to highlight the distribution of phosphorus [18]. Moreover, the architects and the commissioner wished to collect data about the mechanical behaviour of several types of armatures which are sometimes still in place to reuse them in the building structure.

Therefore 4 entire cramps, 4 cresting elements and 1 tie-rod iron sample were taken and machined into tensile tests pieces to measure their true characteristics. The analytical protocol and measurement conditions of these experiments are presented in L'Héritier et al 2019 [19].

Metallographic examinations reveal that most structures are highly ferritic regardless of the armature type (figure 5). Only a few cramps are slightly carburized but none can be considered as steel as they never exceed 0.3 %C in average. On the other hand, two nails are made of homogeneous steel close to the eutectoid with about 0.5-0.7 % C in average. The slag inclusion proportion in the matrix (in average 2-3 % and sometimes up to 10%) is comparable to previous studies on pre-modern construction iron [19,20]. Phosphorus is also present in most samples. Nital etching reveals the presence of ghost structures in more than half the studied samples and Oberhoffer's etching performed shows its heterogeneous distribution at a microscopic and macroscopic scale (figure 5) [21]. Let us recall that high levels of phosphorus create iron embrittlement [22]. Finally, many welds were observed especially in the cramps (figure 5 b), which seem to have been regularly forged by the assembly of several metal pieces. The tensile tests provide the mechanical characteristics of these alloys.

Tensile tests were performed on cylindrical specimens with 8 mm diameter and 64 mm gauge length according to the protocol published by L'Héritier et al. [19]. They were done on an MTS machine with 100 kN capacity controlled by an extensometer with 50 mm gage length and a full range of 10%. All tests were performed at room temperature (300 K) until failure with a constant strain rate of $1.3 \times$ 10-4 sec-1. Young's modulus is comprised between 185 and 215 GPa for all samples, quite well corresponding to that of iron and mild steels [23]. However, other characteristics show a great heterogeneity from one sample to another and sometimes even for two samples of the same object (figure 6 a). The elongation goes from 2,5% to 25% and tensile strength from 280 to 530 MPa revealing characteristics way below those of modern steels. Fractures are usually ductile with slag inclusion inside the dimple, yet some samples present brittle transgranular fracture and brittle intergranular fracture, due to the presence of phosphorus (figure 6 b, c, d). The significant presence of phosphorus, the presence of many non-metallic inclusions, sometimes millimetre-sized, and the numerous welds, more or less well executed, explain the very poor quality of the metal used in the cathedral of Notre-Dame de Paris at the time of its construction and during the restoration work carried out in the middle of the 19th century by Viollet-le-Duc. The relatively modest mechanical properties of the iron and steel used in these periods are not unique to Notre-Dame de Paris and can be found in all the buildings studied earlier in medieval and early modern France (see figure 7) [19] and Italy [24-26]. Only the modern carbon cresting installed in the 20th century have mechanical properties, admittedly among the weakest in the multitude of contemporary steels, but normal for this type of steel in terms of its composition, production and shaping. Finally, no observation revealed any impact of fire on the microstructure and mechanical properties of the various reinforcements studied. These armatures can therefore be reused in full knowledge of these characteristics, provided that they would only work in the elastic domain.



Figure 5: Micrography of some of the studied iron samples. (a) Oberhoffer etching showing microscopic and macroscopic variation of phosphorus content (b) AGR VN2, Nital etching, showing welding lines separating zones with different slag inclusion size and content in a globally ferritic matrix (c) AGR TRIB 01 Nital etching, showing a ferritic structure with embedded slag inclusion and a carburised band (0,4 to 0,7 %C)



Figure 6: Tensile tests on Notre-Dame samples. (a) Tensile true stress/true strain curves of reinforcements. (b) Fracture examination of AG VN4 showing different behaviour on each side of the welding lines. (c) transgranular brittle fracture by cleavage (d) ductile fracture with slag inclusion inside the dimple



Figure 7: Mechanical characteristics of Notre-Dame ferrous reinforcements compared to modern steels and other medieval and modern construction iron on the Ashby Map [27]

4.2 Iron production and provenance

Many questions still arise regarding Notre-Dame's construction. How was the iron produced and transported to the building site? Several iron production processes indeed coexisted in medieval and modern history. In Northern Europe, the traditional bloomery process where iron is directly produced from the reduction of iron ore is predominant since the Iron Age until the end of the Middle Ages. Blast furnaces producing cast iron which can be decarburised into iron in fineries appear in the late 13th and 14th centuries and spread in the North of the French Kingdom in the second half of the 15th century [28]. Both iron production processes were likely to be involved over the long time scale of Notre-Dame's construction and restorations from the 12th to the 19th centuries. Moreover, several iron supplying regions, especially in a site as central as the French capital, could have provided the metal through terrestrial or fluvial means of transportation. It is indeed a known fact for the late Middle Ages that iron was traded all along the Seine, downstream and upstream but local production units accessible from Paris by road existed [29]. First, the analysis of slag inclusions embedded in the matrix of ancient ferrous alloys can provide several information on their provenance and on the technological processes used for their production. Major element analysis of slag inclusion using SEM-EDS is able to discriminate between iron produced in bloomery furnaces and iron coming from the refining of cast iron produced in blast furnaces, which spread in Europe in the end of the Middle Ages [30,31]. Trace element analysis of the same slag inclusions are another key towards the study of iron provenance and supply of the building yard. Comparing trace element ratios as proposed by Leroy et al Disser et al or L'Héritier et al [32–34] can also indicate which iron armatures are likely to come from the same area of production and thus allow reconstructing the building site supply and construction phases as well as the ancient metal trading routes.

Slag inclusion of all previously presented samples were analysed according to the protocol proposed by Dillmann and L'Héritier [30] and adapted by Disser et al [31]. Several hundreds to thousands of SI were analysed per sample by Energy Dispersive Spectrometry coupled Scanning Electron Microscope to have the best representativeness possible of the average produced slag and to get information on the metallurgical processes. Trace element composition was also performed on selected slag inclusions (about 10 per zone of interest in each artefact) by LA-ICP-MS using the IRAMAT-CEB device according to the methodology published by L'Héritier et al [35] in order to question the geographical provenance of these artefacts [16].

The first results on major element composition show a clear distinction between the metallurgical processes used to produce the metal according to the type of sample (figure 8). On the one hand, all the iron cramps (tribunes, nave chapels and upper walls) are made of bloomery iron, which is the only process existing in Europe at the building time of Notre-Dame. This suggests that they are compatible with the medieval construction phases of the building [36,37]. Although their precise dating still has to be performed, because the bloomery process remains in use relatively later in some parts of Europe [38,39], this primary result is an important clue towards the use of iron in the initial design of the monument. On the other hand, all forged nails but two are distinctively located in the finery iron area, meaning that they were forged from iron produced in blast furnaces, consequently later than the initial building time, regardless of their size and typology. Therefore, we can conclude that all iron reinforcements of the framework were undertaken in late restorations phases after the middle of the 15th century when this iron production. Even the nails coming from the framework wooden pathway, which was so far presumably associated with the 13th century construction phases [40], seem to be much later and highlight a modern construction (or restauration) of this ensemble. Only one nail (and maybe a second one located in the undetermined zone) can surely be associated with the more ancient bloomery process (CH14 MCL 03 & VN9 GCL 17). Such a feature still has to be explained. Radiocarbon dating of some of these nails was carried out to understand if they can represent an undocumented former restauration phase or is rather an evidence of iron recycling (see §4.3). Lastly, two different groups can be highlighted within the tie-rods and cresting implemented in the 19th century. Those (tierod + 2 cresting) with inclusions containing high iron oxide silica, calcium and phosphorus contents (in average FeO = 50-75 %, SiO2 = 5-15%, P2O4 = 10-15%, CaO = 10-16 %) are typical of 19th century ferrous alloys, despite the restauration by Mouton in the 2000's, suggesting a reuse of the former cresting. On the other hand, the two cresting elements that Fonquernie had restored display very small slag inclusion with silica and MnS particles as well as an homogeneous carbon distribution in the metallic matrix which is typical of modern steels refined by means of a converter from the end of the 19th century, rather indicating an absence of reuse. The results are in accordance with the conclusion of their historical and typological study [41].



Figure 8: Discrimination of iron reduction processes using slag inclusion composition [30]. Each dot represents one object determined by its slag inclusions average weighted contents. ** refers to the compound average weighted content reported to iron. Grey and white dots constitute a corpus of reference for bloomery and finery iron. Coloured dots are the objects analysed within this study.

4.3 Dating the iron armatures and the construction phases

Dating in archaeology of standing structures is probably the most important problematic in order to understand the construction phases and possibly the progressive implementation of some construction materials in the building throughout these campaigns. Many questions arise in the case of Notre-Dame so as in many buildings. Some crucial dates such only related to the interpretation of a single historical source [37,42]. For example, the completion of the first framework and the vaults of the choir are deduced from the chronicle of Robert de Torigni who claims in 1177 that the church choir is completed "With the exception of its major cover" (*cujus caput iam perfectum est excepto majori tectorio* [43 II, 68]) and the consecration of the main altar in May 1182, interpreted as the achievement of the framework and vaults. The dating of the vaults has for example been recently questioned thanks to an archaeological study of the fallen nave arch [44]. Reinterpretation of dendrochronological results has also led to modifications in the dating of the framework [40,45]. Iron armatures, which are present on every level of the monument, can take part in this reflection.

When they contain some steely parts, ferrous alloys can indeed be dated using radiocarbon dating according to the method developed by Leroy et al 2015 [46]. Actually, the carbon is coming from the smelting stage during which charcoal is used for the reduction. The process occurs at high temperature (about 1300°C) and a part of the carbon from the charcoal diffuses into the metal, creating these carburised zones. The carbon is sampled within these carburized zone, a iron-carbon alloy (up to 0,8 % C for eutectoid steels). Several iron artefacts among all the armatures sampled at Notre-Dame are sufficiently carburized and were dated: 2 cramps from the tribunes, erected in the middle of the 12th c., 4 cramps of the upper walls probably built in the beginning of the 13th c. and 2 nails of the

framework, one made of steel and both identified as made from bloomery process iron (see above). These dating contribute to a better understanding of the implementation of iron at the different stages of Notre-Dame's construction, highlighting the use of iron as early as the middle of the 12th c. construction campaigns [16]. Notre-Dame is now the earliest monument using such iron armatures, almost 40 years before Chartres and Bourges [3,4,46], thus considerably renewing our apprehension of medieval construction techniques. Secondly, they also allow dating the reinforcement of the upper walls to link them with the dates obtained on the framework and later on the mortars. Crossing all these data will provide a better understanding of the monument's chronology. The results obtained on the two nails are presented here (figure 9). The smaller iron nail of the suspended wooden pathway in the choir was dated from the 14th century (1306-1422 AD). Let us recall that all other studied small nails are made of iron from finery and likely to be associated to later works, thus suggesting evidence of reuse. The bigger nail of the nave made of steel is dated from the 16th century (1475-1642 AD). There is however no recollection of major works to the framework before 1681. Do both dating rather indicate recycling practices? Alternatively, could they document unknown consolidations phases of the framework? Other dating on big nails and smaller finery nails should be performed to confirm these first tendencies. The precise role of these big nails still has to be explored through the analysis of the wooden vestiges of the framework, now available for study.



DxCal v4.4.4 Bronk Ramsey (2021); r:5 Atmospheric data from Reimer et al (2020)

Figure 9: Radiocarbon dating of the framework nails CH14 MCL03 (wooden pathway, choir) and VN9 GCL17 (framework, nave). "The radiocarbon result were calibrated with the OxCal 4.4 software [47] using the IntCal20 calibration curve [48]

5. Conclusion

This brief presentation of the undergoing work on Notre-Dame's reveals the extreme intricacy of the historical issues and the questions related to the conservation and restoration of the monument on a large range of topics linked to the use of iron in construction: materials quality, reuse, dating and implementation... All these questions can only be explored through tight and focused interdisciplinary studies as the ones presented in this paper attempt to illustrate. The impetus given by the scientific project on all construction materials will allow an unprecedented collection of these data, in order to understand better the construction of the monument and its structure. We can be sure that the combination of mechanical data and data on provenance and dating obtained on stone, wood, glass, iron and lead, thanks to methodologies developed over several years or decades in the field of heritage materials will be fruitful in renewing our understanding of the building. Yet, the knowledge on iron goes beyond the sole case of Notre-Dame. The blaze also generated new methodological

breakthroughs, which might have further exploitation in the <u>field of archaeology</u> or heritage: thanks to the archaeological survey, original developments linked to the study of corrosion are also currently being carried out to determine the temperatures reached by the irons. Notre-Dame thrives on science, but science thrives on Notre-Dame.

Acknowledgements

The authors would like to thank the CNRS and Ministry of Culture for the general organization and funding of this research as well as all the researchers involved in this program, especially the scientists of Metal Workgroup for very fruitful discussions. They also thank the Service Régional de l'Archéologie, Conservation Régionale des Monuments Historiques, Etablissement Public Rebâtir Notre-Dame de Paris (RNDP) and the Architectes en Chef des Monuments Historiques (P. Villeneuve, P. Prunet, R. Fromont) for the authorizations to access the monument and sample these armatures to perform this study. Many thanks to Jarnias company for their material help in sampling. The authors would also like to thank Philippe Chevallier, Yvan Cotrebil and Rémy Pirès from ICMPE for their assistance in the mechanical tests, the sample preparation and the analyses and experiments carried out in the OM and SEM. The authors thank as well Lise Boutenègre, Emma Daudré and Arthur Poissenot for their contribution to this work. Study of the nails was partly funded by the DIM-MAP. Tensile tests and corrosion studies were partly funded by the Etablissement Public RNDP. At last, many thanks to all colleagues involved in the sorting of archaeological artefacts who made this study possible.

References

- M. L'Héritier, Le fer et le plomb dans la construction monumentale au Moyen Âge, de l'étude des sources écrites à l'analyse de la matière. Bilan de 20 ans de recherches et perspectives, Ædificare. Revue Internationale d'histoire de La Construction. (2019) 79–121.
- [2] A. Timbert, ed., L'homme et la matière : l'emploi du plomb et du fer dans l'architecture gothique. actes du colloque, Noyon, 16-17 novembre 2006, Picard, Paris, 2009.
- [3] E. Lefebvre, M. L'Héritier, De l'emploi du fer dans la structure de la cathédrale de Chartres. Approche qualitative et quantitative, in: Chartres. Construire et Restaurer La Cathédrale (Xle-XXIe s.), Presses universitaires du Septentrion, Villeneuve-d'Ascq, 2014: pp. 287–306.
- [4] M. L'Héritier, Les armatures de fer de la cathédrale de Bourges : nouvelles données, nouvelles lectures, Bulletin Monumental. 174 (2016) 447–465.
- [5] M. Férauge, P. Mignerey, La cathédrale de Bourges et l'utilisation du fer dans l'architecture, Bulletin Monumental. 154 (1996) 129–148.
- [6] M. L'Héritier, P. Dillmann, P. Benoit, Iron in the building of gothic churches: its role, origins and production using evidence from Rouen and Troyes, Historical Metallurgy. 44 (2010) 21–35.
- [7] M. L'Héritier, Réflexion sur les usages du fer à la cathédrale de Coutances et dans l'architecture gothique normande, in: P. Bouet, G. Désiré dit Gosset, F. Laty (Eds.), La Cathédrale de Coutances. Art & Histoire, Orep éditions, Bayeux, 2012: pp. 39–56.
- [8] P. Dillmann, De Soissons à Beauvais : le fer des cathédrales de Picardie, une approche archéométrique, in: L'homme et La Matière: L'emploi Du Plomb et Du Fer Dans l'architecture Gothique. Actes Du Colloque, Noyon, 16-17 Novembre 2006, Editions A&J Picard, 2009: pp. 93– 111.
- [9] J.-L. Taupin, Le fer des cathédrales, Monumental. (1996) 18–27.
- [10] M. L'Héritier, Fer et plomb sur le chantier, in: Saint-Denis. Dans l'éternité Des Rois et Des Reines de France, La Nuée bleue, Strasbourg, 2015: p. 96.
- [11] A. Erlande-Brandenburg, L'architecture rayonnante et le métal, Documents d'archéologie. 46 (1996) 18–27.

- [12] B. Fonquernie, G. Françoise, B. Mouton, M. Viré, Les matériaux mis en œuvre, in: Notre-Dame de Paris, La Nuée bleue, Strasbourg, 2012.
- [13] E.-E. Viollet-le-Duc, Dictionnaire raisonné de l'architecture française du XIe au XVIe siècle, Morel, 1854.
- [14] J.-M. Leniaud, Jean-Baptiste Lassus (1807-1857) ou le temps retrouvé des cathédrales, Droz, Genève, 1980.
- [15] T. Zimmer, D. Chaoui-Derieux, L. Leroux, B. Bouet, A. Azéma, D. Syvilay, E. Maurin, F. Mousset, From debris to remains, an experimental protocol under emergency conditions, Journal of Cultural Heritage. (2023). https://doi.org/10.1016/j.culher.2022.12.009.
- [16] M. L'Héritier, A. Azéma, D. Syvilay, E. Delqué-Kolic, L. Beck, I. Guillot, M. Bernard, P. Dillmann, Notre-Dame de Paris: The first iron lady? Archaeometallurgical study and dating of the Parisian cathedral iron reinforcements, PLOS ONE. 18 (2023) e0280945. https://doi.org/10.1371/journal.pone.0280945.
- [17] G. Vander Voort, Microstructure of ferrous alloys, in: Analytical Characterization of Aluminum, Steel and Superalloys, 2001: pp. 157–234.
- [18] J.W. Stewart, J.A. Charles, E.R. Wallach, Iron-phosphorus-carbon system: Part 2– Metallographic behaviour of Oberhoffer's reagent, Materials Science and Technology. 16 (2000) 283–290.
- [19] M. L'Héritier, I. Guillot, P. Dillmann, Microstructural Characterization and Mechanical Properties of Iron Reinforcements in Buildings from the Medieval and Modern Periods in France, International Journal of Architectural Heritage. 13 (2019) 507–519. https://doi.org/10.1080/15583058.2018.1563229.
- [20] M. L'Héritier, P. Dillmann, S. Aumard, P. Fluzin, Iron? Which iron? Methodologies for metallographic and slag inclusion studies applied to ferrous reinforcements from Auxerre Cathedral, in: J. Humphris, T. Rehren (Eds.), The World of Iron, 2013: pp. 409–420.
- [21] J.W. Stewart, J.A. Charles, E.R. Wallach, Iron-phosphorus-carbon system. Part 3 Metallography of low carbon iron-phosphorus alloys, Materials Science and Technology. 16 (2000) 291–303.
- [22] J.W. Stewart, J.A. Charles, E.R. Wallach, Iron-phosphorus-carbon system Part 1 Mechanical properties of low carboniron-phosphorus alloys, Materials Science and Technology. 16 (2000) 275–282.
- [23] J. Bénard, A. Michel, J. Philibert, J. Talbot, Métallurgie générale, 2e éd. édition, Masson, Paris ; New York, 1984.
- [24] C. Calderini, P. Piccardo, R. Vecchiattini, Experimental Characterization of Ancient Metal Tie-Rods in Historic Masonry Buildings, International Journal of Architectural Heritage. 13 (2019) 425–437. https://doi.org/10.1080/15583058.2018.1563230.
- [25] M. Bellanova, A. Baggioli, B. Rivolta, R. Felicetti, Metallurgical characterisation of a historical metal tie-rod from Milan Cathedral, Construction and Building Materials. 226 (2019) 888–898. https://doi.org/10.1016/j.conbuildmat.2019.07.272.
- [26] P. Matteis, G. Scavino, Mechanical Performance of Historical Wrought Iron Rods, Archaeometry. (2019). https://doi.org/10.1111/arcm.12470.
- [27] M.F. Ashby, Materials Selection in Mechanical Design, 5e édition, Butterworth-Heinemann Ltd, Amsterdam ; Cambridge, MA, 2016.
- [28] P. Dillmann, M. L'Héritier, Archéométrie et histoire des techniques : les procédés direct et indirect en sidérurgie (XIVe-XVIIe siècle), Artefact. (2016) 63–81.
- [29] M. Mollat, R.-H. Bautier, Le trafic fluvial sur la Seine au pont de Meulan au milieu du XVe siècle, Bulletin Philologique et Historique Du Comité Des Travaux Historiques et Scientifiques. (1959) 251–296.
- [30] P. Dillmann, M. L'Héritier, Slag inclusion analyses for studying ferrous alloys employed in French medieval buildings: supply of materials and diffusion of smelting processes, Journal of Archaeological Science. 34 (2007) 1810–1823.
- [31] A. Disser, P. Dillmann, C. Bourgain, M. L'Héritier, E. Vega, S. Bauvais, M. Leroy, Iron reinforcements in Beauvais and Metz Cathedrals: from bloomery or finery? The use of logistic

regression for differentiating smelting processes, Journal of Archaeological Science. 42 (2014) 315–333.

- [32] A. Disser, P. Dillmann, M. Leroy, M. L'Héritier, S. Bauvais, P. Fluzin, Iron Supply for the Building of Metz Cathedral: New Methodological Development for Provenance Studies and Historical Considerations, Archaeometry. 59 (2017) 493–510. https://doi.org/10.1111/arcm.12265.
- [33] S. Leroy, S.-X. Cohen, C. Verna, B. Gratuze, F. Téreygeol, P. Fluzin, L. Bertrand, P. Dillmann, The medieval iron market in Ariège (France). Multidisciplinary analytical approach and multivariate analyses, Journal of Archaeological Science. 39 (2012) 1080–1093.
- [34] M. L'Héritier, P. Dillmann, G. Sarah, Deciphering the Iron Provenance on a Medieval Building Yard: The Case of Bourges Cathedral, Minerals. 10 (2020) 1131. https://doi.org/10.3390/min10121131.
- [35] M. L'Héritier, S. Leroy, P. Dillmann, B. Gratuze, Characterisation of slag inclusion in iron objects, in: Recent Advances in Laser Ablation ICP-MS for Archaeology, Springer, 2016.
- [36] C. Bruzelius, The Construction of Notre-Dame in Paris, The Art Bulletin. 69 (1987) 540–569.
- [37] D. Sandron, Notre-Dame de Paris. Histoire et archéologie d'une cathédrale (XIIe-XIVe siècle), Cnrs, 2021.
- [38] C. Verna, Le temps des moulines: fer, technique et société dans les Pyrénées centrales, XIIIe-XVIe siècles, Publications de la Sorbonne, 2001.
- [39] M. Leroy, Autour de la « minette » : la perduration de la production de fer en bas fourneau en Lorraine à la fin du Moyen Age, in: L'innovation Technique Au Moyen Age: Actes Du VIe Congrès International d'archéologie Médiévale, Errance, Paris, 1998: pp. 145–150.
- [40] F. Epaud, Etude archéologique de la charpente de Notre-Dame de Paris, 2020.
- [41] D. Syvilay, A. Azéma, Cathédrale Notre-Dame 75, Paris. Crêtes et épis de faîtage. Armatures en fer Etude, inventaire et typologie, 2020.
- [42] Cardinal André Vingt-Trois, Notre-Dame de Paris, La Nuée bleue, Strasbourg, 2012.
- [43] L. Delisle, Chronique de Robert de Torigni, abbé du Mont-Saint-Michel, 1872. https://gallica.bnf.fr/ark:/12148/bpt6k36182g (accessed November 25, 2021).
- [44] C. Moulis, A. Ybert, B. Phalip, The fallen arch of Notre-Dame. A reassessment of its chronology in light of new archaeological data, Journal of Cultural Heritage. (n.d.).
- [45] F. Epaud, La charpente de Notre-Dame de Paris : état des connaissances et réflexions diverses autour de sa reconstruction, Scientifiques de Notre-Dame. (2019). ffhal-03176917f,.
- [46] S. Leroy, M. L'Héritier, E. Delqué-Kolic, J.-P. Dumoulin, C. Moreau, P. Dillmann, Consolidation or initial design? Radiocarbon dating of ancient iron alloys sheds light on the reinforcements of French Gothic Cathedrals, Journal of Archaeological Science. 53 (2015) 190–201.
- [47] C.B. Ramsey, Bayesian Analysis of Radiocarbon Dates, Radiocarbon. 51 (2009) 337–360. https://doi.org/10.1017/S0033822200033865.
- [48] P.J. Reimer, W.E. Austin, E. Bard, A. Bayliss, P.G. Blackwell, C.B. Ramsey, M. Butzin, H. Cheng, R.L. Edwards, M. Friedrich, The IntCal20 Northern Hemisphere radiocarbon age calibration curve (0–55 cal kBP), Radiocarbon. 62 (2020) 725–757.
- [49] M. Bouchar, E. Foy, D. Neff, P. Dillmann, The complex corrosion system of a medieval iron rebar from the Bourges' Cathedral. Characterization and reactivity studies, Corrosion Science. 76 (2013) 361–372.
- [50] A.R. Dinesen, C.T. Pedersen, C. Bender Koch, The Thermal Conversion of Lepidocrocite (γ-FeOOH) Revisited, Journal of Thermal Analysis and Calorimetry. 64 (2001) 1303–1310. https://doi.org/10.1023/A:1011582004634.
- [51] R.Y. Chen, W.Y.D. Yeun, Review of the high-temperature oxidation of iron and carbon steels in air or oxygen, Oxidation of Metals. 59 (2003) 433–468.
- [52] S. Gialanella, F. Girardi, G. Ischia, I. Lonardelli, M. Mattarelli, M. Montagna, On the goethite to hematite phase transformation, Journal of Thermal Analysis and Calorimetry. 102 (2010) 867– 873.
- [53] J. Paidassi, Contribution à l'étude de l'oxydation du fer dans l'air dans l'intervalle 700-1250° C, Revue de Metallurgie. 54 (1957) 569–585.