Microstructural characterization of two Koto age Japanese swords

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Two Japanese long blades of the Ancient Sword (Koto) age have been analysed through time of flight neutron diffraction. This technique allows the determination of several microstructural properties on different size gauge volumes. The results of the experiment provided a quantitative multiphase characterization of the steel composition of the blades and the determination of peculiar properties of the material, such as the texture, the strain level and the grain size of the crystallites.

Keywords:
steel; difractometry; physical metallurgy ; history of metallurgy; materials characterization

INTRODUCTION

Japanese swordsmaking is probably one of the best examples of skills in historical metallurgy field in the history of mankind. A semi-empirical approach has been able to optimize the metallurgical characteristics of the components of the blade in order to produce the best results for every single element of the sword. Neglecting the human manpower cost, if we consider that the production of high carbon steel was an expensive procedure in terms of energy and materials, the use of the materials has been optimized since the high carbon parts have been used only where strictly necessary and the quality of the low and high carbon steel was assured by the smithing process.

Several research results have been already published on the steel production and the forging processes [1-6]. Here, we would like to focus on the description of the manufacturing procedure of those parts that have been further investigated.

The iron ore sand was reduced in the tatara furnace through a long residence time and the resulting material is a bloom containing slags and portions of metal with low and high carbon content. The bloom was then reduced in pieces by hammering and, according to the colour, the lumps were divided into groups having different carbon content. This is the first step where the experience is mandatory since to tell the carbon content in a lump from the colour is a difficult task. After that, the steel was moved to the swordsmaker workshop. We remind that the lumps are still full of slag inclusions, due to the smelting process, since the liquid state is never reached in the process. In order to reduce the slags, by decreasing their size, and partially remove them, the lumps with low or high carbon content were placed on an iron plate and hammered to form a sheet. The sheet was then cut in the middle, superimposed, and hammered again to form a new sheet that now contains two layers of the tatara furnace steel. The process is repeated several times and this method is able to remove slag inclusions and to reduce the rest to micrometric or even submicrometric dust. The number of hammering and folding is not constant and depends on forging tradition as well as on the function of the worked steel inside the artefact [7]. The parts designed to form the cutting edge, the external skin, and sometimes the back of the blade are folded between 10 and 16 times while the core of the blade and the tang are folded between 4 and 6 times since the presence of residual slag inclusions to a larger extent is not too damaging in these parts.

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This process has anyway two consequences: the reduction of the average carbon content in the sheet of steel due to the oxidation during the heating and the formation of thin layers of goethite between the layers during every folding process.

Once all the sheets have been prepared, they are assembled to form the blade by welding them together through mechanical action. The core and the tang are formed by the low carbon steel while the external sides, the edge and sometimes the back are formed by one or more different sheets of medium/high carbon steel.

The blade is then shaped in order to have it with one edge and slightly curved as a sabre. It is then covered with charcoal containing clay with different thicknesses (not a good thermal insulator but just a poor conductor) with the thinnest coverage on the edge. The blade is then warmed up and quenched. At this point the blade is fully hardened on the edge and in the part immediately below it and it is also partially hardened through a transition to bainite on the other external parts covered by a thin layer of clay. The transition to martensite in the edge is also responsible for the increase of the curvature and this effect keeps the blade in a permanent tensile state able to partially balance the sudden strain action brought by blows.

All this information comes out from metallographic analysis taken on a few samples cut and studied in the past years [8-10]. It is anyway difficult to take a similar approach on a large scale research program since a reliable analysis would need the full cut of the blade in more than one slice in order to obtain a full reconstruction of the microstructure.

A different and less invasive approach is therefore mandatory and the application of high resolution diffraction techniques can

FIG. 1 ***Pictures of the Bizen Sue-Aoe tachi blade (upper) and of the Mino Kanesada katana blade. It is evident the presence of some holes in the tang testifying the modification of the original length. The curvature of the two swords is also very different. The directions of the strains measured on ENGIN-X are also shown.***

Fotografia della lama di tipo tachi di tradizione Bizen e scuola Sue-Aoe (in alto) e della lama di tipo katana di tradizione Mino e scuola Kanesada. E' evidente la presenza di numerosi fori nell'impugnatura che indicano modifiche rispetto alla lunghezza originaria. Nell'immagine sono riportate anche le direzioni delle deformazioni residue misurate sul diffrattometro ENGIN-X.

be an answer. Time of Flight Neutron Diffraction (ToF-ND) is outstanding in terms of diffractometric approaches since it is able not only to quantify the relative amount of the crystalline phases, but also to give information on the microstructural state measuring the texture level and directions, the residual strains in selected phases and the size of the grains. The results obtained through this technique are superimposed only partially to the ones obtained by conventional metallography. The ideal approach would be the application of both methods, however destructive techniques can not be applied to valuable artefacts Nevertheless ToF-ND technique alone is able to give a satisfactory amount of results which can provide a full characterization of the status of the studied sample.

ToF-ND furnishes information on the sample as it is with no need of surface treatment or sampling. The measured properties are representative to an average volume which can vary from a few mm^3 in size up to a few cm^3.

SAMPLES

Our study focused on two Japanese long swords of the Koto (Ancient sword) age [3,4] in order to determine the composition of the steel, the presence of phases related to the smelting (fayalite, wuestite, troilite) and the presence of oxides due to the mineralization (goethite, magnetite, hematite). All these measurements were carried out on the Italian Neutron Experimental Station (INES) diffractometer located at the ISIS pulsed neutron source [11]. On the same instrument, the ferrite phase has been also investigated in order to determine in different parts of the blade (in height and in position from the ridge to the edge): the texture level, the strain level, and the grain size.

A careful strain distribution analysis has been also carried out using the ENGIN-X engineering beamline at ISIS [12]. The two chosen swords, shown in fig. 1, pertain to two different traditions of the Koto Age: namely the Bizen and the Mino. The former developed as one of the two most ancient and was born in the southern part of Honshu island while the latter developed in the Mino province. Both the areas are rich of iron ore mines and developed under the protection of local nobles. Both the Mino and Bizen traditions were very high in production and, during the warring state period, there were many mass production blades done according to their forging procedure. Both the blades are not signed and their attribution is done through stylistic analysis which comprehends visual inspection of the most significative peculiarities of the blades: the shape of the tip, the cross section, the modulation of the curvature along the sword, the shape of the tang, the shape of the edge and of the ridge and, moreover, the grain of the surface and the shape of the temper line (hamon).

The Bizen blade is a long sword used on horseback (tachi) produced by the Aoe school in the province of Bitchu at the end of 15th century. The presence of four holes in the tang testifies it has been deeply shortened to reach its actual length. The curvature is low with a higher curvature in the lower part of the blade and an almost straight blade close to the tip. The thickness and the size from the edge to the ridge are reduced in comparison with the original ones since the blade has been polished many times during its life. The traditional polishing method consists in grinding the surface with seven stones with decreasing coarseness. This process removes about 100-200 microns of thickness normal to the surface so that it corresponds to a few mm in the ridge-edge direction. The last polishing has been done 10 years ago and the surface is in ideal conditions for a stylistic analysis with the hamon line perfectly visible even if weakened in contrast by the many polishing.

Tha Mino blade is an infantry long sword (katana) produced by the Kanesada school in the province of Mino at half of the 16th century. The tang is the original one so no shortening seems to be ever been applied. The curvature is high and constant for the whole length of the blade.

EXPERIMENTAL SET-UP AND MEASURING POINTS

The INES and ENGIN-X diffractometers present complementary characteristics. Both can be used for quantitative multiphase analysis but the highly specialized nature of ENGIN-X additionally provides the residual strain measurements in the small gauge volume. The quantitative analysis has been hence applied on INES together with the peak shape, texture and grain size analysis thanks to the high resolution of the backscattering banks ($\delta d/d \approx 0.1\%$).

The samples have been gently clamped in an aluminium frame holder and put inside the experimental tank of the instrument. Thanks to the specifically designed alignment neutron imaging device [13] the measuring gauge volumes have been selected and are reported in Table I for the two blades. The number of points and measuring time for each of them has been chosen by combining the available beam time (6 days) devoted to this ex-

Sample	Tip	Monouchi				Half blade				Tang
		average	edge	core	ridge	average	edge	core	ridge	
Aoe	X	X	X	X	X					X
Kanesada		X	X		X	X	X	X	X	X

TAB. I ***Measured points of the two swords on the INES diffractometer. Monouchi is the upper part of the blade close to the tip, usually used to strike blows.***

Punti misurati nelle due lame tramite il diffrattometro INES. Il monouchi è la parte superiore della lama, vicino alla punta, usata solitamente per colpire.

periment with the necessity to reach a signal to noise ratio good enough to detect the weakly scattering mineralization and smelting phases . The average time has been selected to be about 8 hours per point.

The residual strain maps, measured on the ENGIN-X diffractometer, have been taken on several measuring points with the gauge volume of 2x2x2 mm^3. The unstrained reference point "a_0" was selected in the core of the tang , as we assumed that this part was the least affected by mechanical and thermal treatments. The points in which the strain has been measured are four cross sections in the swords at 1/4, 3/8, 1/2 and *monouchi* heights in the blade. Additionally full scan on the edge and the ridge along the blades have been performed. The strains have been measured as $\varepsilon=(a-a_0)/a_0\times10^3$ (with a and a_0 the measured lattice parameter of the strained and reference parts of the sample) and labelled as "millistrain" adimensional units. The selected directions in which strains have been measured are: 1) along the blade from the tang towards the tip (axial (A)), 2) from ridge to edge (transversal (T)) and 3) from one side to the other (normal (N)).

In order to take this measurements the samples have been mounted in a frame containing 6 reference points for the alignment and then through the use of a 3D laser scanner and of the SSCANSS software [14] the frame has been positioned in an electronically controlled x-y-z-ω plate inside the experimental hall so that the measuring positions were known with an accuracy better than 50 μm.

Three components of strain in each point within the samples have been measured. The ENGIN-X setup allowed two directions to be measured at the same time to maximize the efficient use of the instrument. First, the blades were oriented horizontally to measure A&N directions and then vertically to measure T&N directions. The measuring time has been chosen to be 15 and 3 minutes for the two orientations, respectively. This was possible because we could optimize the gauge volume for T&N directions where vertical dimension was extended to 10 mm to provide a five times bigger gauge volume (2x2x10 mm^3). The available beam time for the whole measurements turned out to be 2 days.

ANALYSIS AND RESULTS

The obtained data were processed through Rietveld refinement using the GSAS code [15]. For what concerns the INES data, all the multiphase analysis data have been already published [16,17] and will not be reported here. The results concerning the strain index, texture and grain size of the average measurements have been published as well [18] but will be shown here for sake of clarity. For the ENGIN-X residual strain data, part of them has been published [16, 17] so we will focus on the original data of the edge measurements.

The analysis of the peaks of the high resolution diffractograms taken on the INES backscattering bank can give detailed information on several microstructural properties related to some specific parameters of the function describing the shape of the peak. Specifically the texture level, the strain index and the grain size can be determined through the quantification of the J, S400, and Gam2 parameters respectively for peak profile 4 [19].

Texture is a phenomenon related to solids in which the crystal grains are not isotropically distributed as in powders but are oriented at different extents according to the mechanical work and thermal history of the sample. For example, a heavily cold worked metal sheet will have grains preferentially oriented along the rolling direction. The best way to represent texture is a 2D pole figure in which texture intensity and directions are shown. In order to obtain such a result, diffraction patterns with scattering angles covering the whole solid angle around the sample need to be measured. With a single measurement it is possible to estimate a parameter indicating how much oriented the grains are along a specific direction that is the bisecant the primary and the scattered neutron beam. The parameter is the texture index usually labelled as J. It represents the ratio between the volume occupied by all the grains divided by the volume occupied by isotropic grains. Its value changes from 1 for ideal isotropic grains to infinity for a huge single crystal. Average typical values for heavily worked steel are around 1.2.

The strain is an adimensional index of the inhomogeneity level induced by defects. Unless determining the lattice parameter difference between strained and unstrained parts of a sample, it is not possible to reliably quantify its amount. The strain index determined through peak shape analysis is hence only semiquantitative and gives a relative scale between less and more strained samples. The S400 parameter is directly linked with the anisotropic distribution of the lattice parameter values [20] and indirectly linked through a very complex numerical relation with the strain parameter. This numerical relation is not perfectly known so that it is not possible to determine strain from S400 parameter but it is known that it is monotonic, hence a larger value for S400 indicates a larger strain value.

The grain size parameter (S) is the size of the average defect free diameter for a specific phase and is determined through the value of the secondary Lorentzian width of the diffraction peaks and labelled as Gam2 (γ_2). The grain size is hence not the dimension of the typical grains measured through metallographic analysis but a smaller value directly related to the microstructural diffraction properties. The relationship relating S to Gam2 is:

$$S = \frac{DIFC \times KS}{\gamma_2} \qquad (1)$$

where DIFC is again the ToF/d-spacing conversion parameter and KS is the Scherrer constant (0.8 adimensional).

Every instrument has an upper grain size limit for the validity of the simple relation #1. The limit is related to the scattering angle and other instrumental parameters and the calculated value for INES is around 120 nm so that a certain determination for grain size of phases whose S value is lower than 100 nm can be assumed. This means that only heavily worked or quenched materials can be estimated.

The results for texture (J) strain index and grain size (S) are shown in fig.s 2,3 and 4 respectively.

The data relative to the texture index show a very high value for the tip of the Aoe blade thus indicating a strong work of re-shaping in order to give it its final appearance. The other very high value is relative to the ridge of the Kanesada at half blade. A possible explanation is that a restoration applied in recent times to recover some bending has been done as cold working in order to avoid to ruin the hardening and the surface of the blade so that a texture appeared. A pole figure reconstruction would give a definitive answer to this observed fact. All the other measured points appear to have small texture and the Aoe blade is always less texturized than the Kanesada blade so that the former was shaped at a lower extent than the latter one to give its definitive shape.

The strain index measurements, shown in fig.3, indicate that in the Aoe sword the average strain is constant in the blade and has a small value in the tang. For what concerns the Kanesada sword, the average strain index is constant everywhere but the value is strongly changing between edge, core and ridge, with the maximum value always located in the edge part and the minimum in the ridge. The tang is the least strained part together with the ridge in the lower part of the blade.

Regarding the grain size data (fig.4), only the data below the upper reliability limit have been shown (about 100-120 nm). It is evident that the Aoe sword presents a small size of the grains in all its upper parts (tip and monouchi, edge, core and ridge) having so a resistant microstructure on the whole. The smallest grains are in the edge (35 nm). The Kanesada blade, on the contrary, always has large grains in the ridge but the average values are comparable with the one of the Aoe sword (around 60 nm for both). The grain size in the edge is instead smaller (15 nm). Apart all the considerations about the overpolishing of the Kanesada blade, it seems that the quenching has been done with a quicker temperature drop than in the Aoe case. There might be three different explanations all of them plausible: 1) the thickness of the clay in the edge was thinner (or even absent) in the Kanesada blade than in the Aoe; 2) the temperature of the quenching water was lower in the Kanesada than in the Aoe; 3) the Aoe sword was slightly tempered after quenching.

Going to the ENGIN-X data regarding the strain profiles on the edge, the results of the values for axial, transversal and normal directions are shown in fig.s 5, 6 and 7 respectively. It is evident a change from negative strain values close to the tang to positive values in the upper parts of the swords along the axial direction (fig.5). The Aoe blade has a less pronounced change that is instead very abrupt in the Kanesada one. This fact is induced by the quenching of the blade that is responsible for a modification of the curvature. The upper part is the one usually employed to strike blows which apply a pulsed force that is positive along the axial direction and negative along the transversal one. Since the residual strain distribution is already positive (tensile) for both blades in the axial direction, the structure is in a permanent tensile state in a direction that is opposite to the one induced by the pulsed force of the blow. In this way the resilience of the edge along the axial direction is enhanced. This phenomenon, was induced by quenching. The

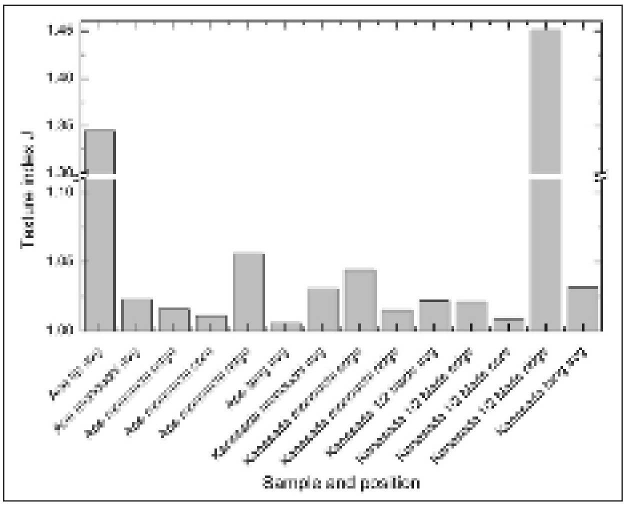

FIG. 2 ***Texture index J for the analysed samples and positions.***

Indice di tessitura J dei vari punti dei campioni analizzati.

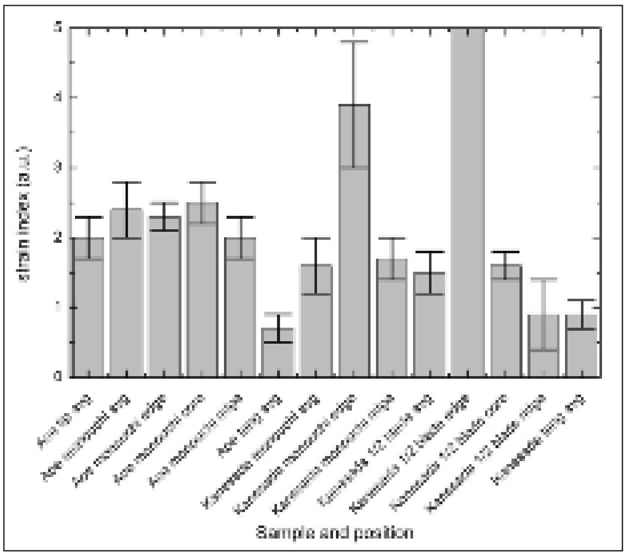

FIG. 3 ***Microstrain values for the samples and positions.***

Valori delle deformazioni residue in funzione dei campioni e delle posizioni.

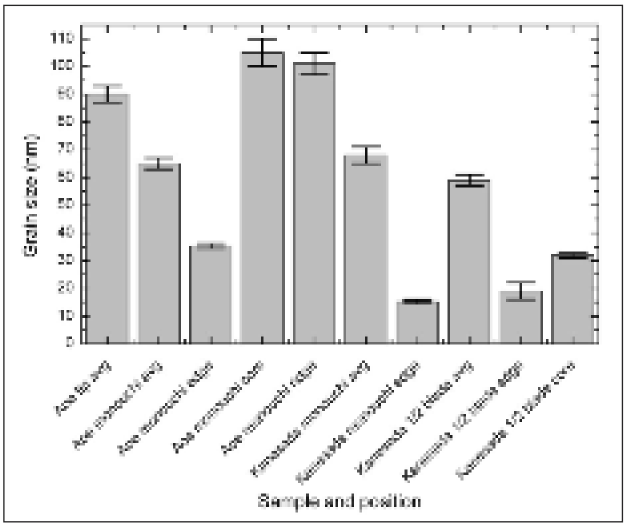

FIG. 4 ***Grain size for the positions where it is smaller than the reliability limit of INES (see text).***

Dimensioni dei domini cristallini nelle posizioni in cui il valore è più piccolo del limite superiore di affidabilità di INES (vedi testo).

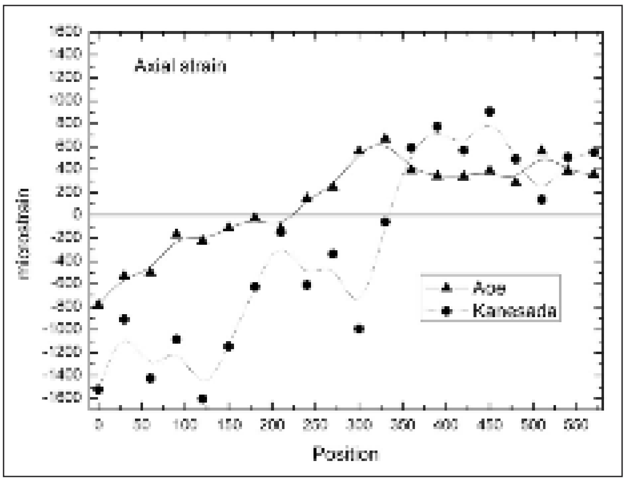

Fig. 5 ***Comparison of axial microstrain in the edge of the two blades. There is a homogeneous trend with negative values close to the tang and positive in the monouchi area.***

Confronto delle microdeformazioni residue assiali sul filo delle due lame. C'è una tendenza omogenea del parametro ad assumere valori negativi in prossimità dell'impugnatura e positivi nell'area del monouchi.

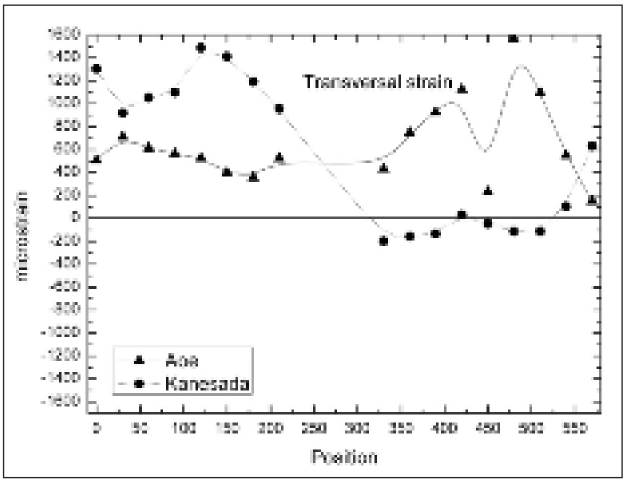

FIG. 6 ***Comparison of the transversal microstrain in the edge of the two blades. The values are constant to a small positive (tensile) value for the Kanesada blade and positive (tensile) close to the tang and negative (compressive) in the monouchi area for the Aoe blade. Note that the trend is exactly opposite the axial direction.***

Confronto fra le microdeformazioni residue trasversali sul filo delle due lame. I valori sono costanti e pari a un valore positivo molto basso (tensione) per la lama Kanesada mentre sono positivi (tensione) in prossimità dell'impugnatura e negativi (compressione) nell'area del monouchi per la lama Aoe. Si noti che la tendenza è esattamente opposta a quella misurata nella direzione assiale.

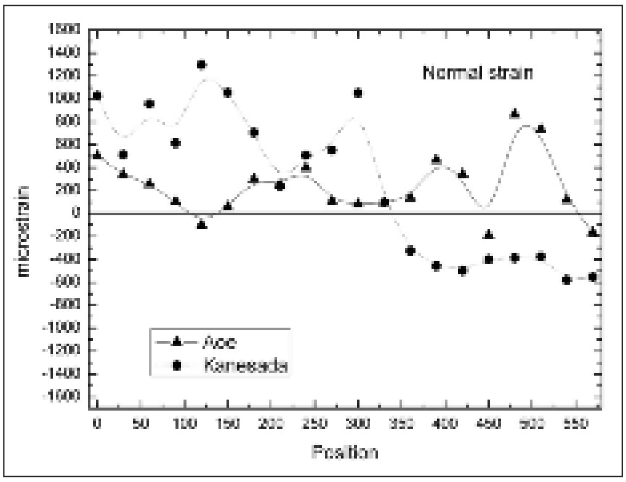

FIG. 7 ***Comparison of the normal microstrain in the edge of the two blades. The values are constant to a small positive (tensile) value for the Kanesada blade and positive (tensile) close to the tang and negative (compressive) in the monouchi area for the Aoe blade. Note that the trend is exactly opposite the axial direction.***

Confronto fra le microdeformazioni residue normali sul filo delle due lame. I valori sono costanti e pari a un valore positivo molto basso (tensione) per la lama Kanesada mentre sono positivi (tensione) in prossimità dell'impugnatura e negativi (compressione) nell'area del monouchi per la lama Aoe. Si noti che la tendenza è esattamente opposta a quella misurata nella direzione assiale.

improved mechanical characteristics of the blade were probably achieved by a try and error process. The strain profiles in the other two directions (transversal and normal) behave exactly in the opposite way as the axial one and this is induced by the elasticity of the material since an expansion in one direction usually implies a compression in at least another one. A negative or low positive value of the transversal strain in the monouchi area (see fig. 5) is an added value as it also increase resilience since the pulsed force is again applied in the same direction.

DISCUSSION AND CONCLUSION

The microstructural properties of two Koto age Japanese long blades have been determined through Time of Flight Neutron Diffraction. The average and punctual properties of texture, strain and grain size have been measured and compared by exploiting the peak shape analysis of the ferrite phase of the data acquired on the INES diffractometer. The residual strain distribution have been mapped on the edge of both blades with high level of details. . The results show a highly efficient and effective forging procedure for both blades , since the properties of the studied parts appear to be suitable to their mechanical functions. The residual strain analysis of the edge of the blades indicates that the Aoe blade was forged with a better knowledge on how to optimize the resilience as it has more favourable distribution than the Kanesada blade.

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Abstract

Caratterizzazione microstrutturale di due lame Giapponesi del periodo Koto

Parole chiave:
acciaio; difrattometria; metallurgia fisica; storia della metallurgia; caratterizzazione materiali

Due spade lunghe Giapponesi del periodo della Spada Antica (Koto) sono state analizzate tramite diffrazione di neutroni in tempo di volo. Questa tecnica consente di determinare alcune delle proprietà microstrutturali su volumi campione di diversa ampiezza. I risultati dell'esperimento hanno fornito la composizione quantitativa delle fasi costituenti le lame. Inoltre hanno consentito di determinare alcune delle proprietà strutturali del materiale come la tessitura, il livello di deformazioni residue e le dimensioni dei domini cristallini.