Microstructure, mechanical behavior, and crystallographic texture in a hot forged dual-phase stainless steel

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Abstract
In this work, the hot forging behavior of a dual phase stainless steel in the temperature range of 850–1250 °C was investigated. The study revealed the occurrence of a significant cracking phenomenon for processing temperatures below 950 °C that was attributed to the combined effect of intermetallic precipitation and severe deformation. EBSD examination highlighted the occurrence of continuous dynamic recrystallization in both ferrite and austenite microstructures for processing temperatures above 1050 °C. Increasing the hot forging temperature to 1250 °C increased the low angle grain boundaries fraction and lowered the one of the high angle grain boundaries. This was accompanied by a gradual change in the crystallographic texture of the material. The mechanical behavior investigation showed that the steel plasticity, sharply dropped after forging at 850 °C, was gradually recovered after hot forging at temperatures above 1050 °C. This was confirmed by nanoindentation measurements that revealed a remarkable increase of the hardness and Young’s modulus of the steel after hot forging at 850 °C and 950 °C due to the dislocation nucleation and the \(\sigma\) phase precipitation at \(\gamma/\delta\) interface. The enhancement of dislocation movement at the vicinity of the grain boundaries in the temperature range of 1050–1250 °C improved the global mechanical properties of the hot forged steel.

Keywords Duplex stainless steel · Hot forging · Microstructure · Mechanical behavior · Crystallographic texture

1 Introduction
During industrial fabrication processes, metallic materials undergo various microstructural transformations due to the thermal, mechanical, or thermomechanical effect of the process. The resulting microstructural changes within the material often constitute a source of stress/strain incompatibilities that can cause its damage. Among the wide range of metallic materials, duplex stainless steels (DSS) are characterized by a dual phase microstructure constituted of austenite (\(\gamma\)) and ferrite (\(\delta\)). Their combination of high mechanical properties and improved corrosion resistance makes them excellent choices for applications in several industrial domains such as petroleum, gas, and petrochemical industries [1–5]. In order to fabricate some specific components, DSS are hot processed by rolling or forging. However, particular attention should be given to the hot processing of these materials due to the complex microstructural evolutions that can take place such as precipitation of intermetallic phases if the deformation temperature is not well controlled. Indeed, different evolutions of the hardening state have been reported in austenite and ferrite during hot processing of DSS [1, 2]. The ferrite is often reported to soften during hot forging by dynamic recovery (DRV), whereas dynamic recrystallization (DRX) is reported to occur preferentially in austenite [6, 7]. Both DRV and DRX intensities are related to the stacking fault energy (SFE) that controls the ability of dislocations to rearrange by cross-slip or by climb. [4–8]. More generally, since austenite and ferrite phases have different elastic and plastic properties in addition to different thermal expansion coefficients, they should act differently during a deformation process. The microstructural and associated hardening evolutions that take place during the hot deformation of single phase austenitic and ferritic stainless steels have been extensively investigated in several research works [7–12]. It was already concluded that easy dislocation
annihilation and rearrangement, leading to dynamic recovery, occurs in ferrite. On the other hand, the low stacking fault energy of austenite weakens the dislocation mobility and limits the occurrence of DRV. Consequently, DRX occurs once a critical strain constituting a driving force for the nucleation of new grains is achieved. Nevertheless, some researchers [4] mentioned that even for materials having high SFE, it was possible to create some experimental conditions that could delay DRV and favor the occurrence of DRX. The situation becomes even more complex when the two phases are deformed simultaneously like in a dual-phase stainless steel due to a non-uniform deformation or stress repartition between its constituent phases. Cizek and Wynne [4] revealed the occurrence of extended DRV in both phases of a DSS hot deformed by torsion. Their results are in agreement with those published by Balancin et al. [13] who also highlighted the dissimilarity of the plastic properties of a hot deformed DSS. Later, Duprez et al. [14] studied the flow behavior of a hot deformed DSS and observed an enhanced ductility that was correlated to the amount of dynamic softening caused by the DRX of austenite. On the other hand, Iza-Mendia [6] reported the suppression of DRX during hot processing of DSS and confirmed the dependence of its mechanical behavior on the initial microstructure. Similarly, DRX was observed by Dehghan-Manshadi et al. [15], whereas Fan et al. [16] demonstrated the occurrence of only DRV in austenite and DRX in ferrite of an as-cast hot deformed DSS. The effect of the crystallographic texture developed during the thermomechanical processing of DSS on their final properties was also investigated in the literature. Ul-Haq et al. [17] reported that the ferrite crystallographic texture formed by hot rolling in a DSS consisted of α fiber, whereas the austenite one contained copper and brass components (see Section 3 for the description of preferred orientations). For Padilha et al. [18] and Cizek et al. [19] however, the austenite texture in a hot processed DSS appears to be dominated by the cube and brass components. Patra et al. [20] investigated the crystallographic texture evolution during thermomechanical processing of a lean DSS. They pointed out that the ferrite was dominated by the cube and rotated cube components, whereas austenite was mainly characterized by deformation textures such as copper, brass, and rotated Goss. Recently, Moura et al. [21] concluded that both ferrite and austenite exhibit texture heterogeneities due to the complexity of the thermomechanical process.

As it can be seen, the literature reports a great number of research works dealing with the hot deformation behavior of DSS such as hot rolling, hot torsion, and compression. It should be mentioned though that the observed softening mechanisms and texture evolutions are still quite dispersed and sometimes contradictory, which tends to underline the important role of the initial microstructure and of some critical parameters of the thermomechanical processing on the resulting characteristics of the material. On the other side, and in spite of its industrial importance, the hot forging behavior of DSS is much less documented. In piping industry, for example, this process is widely applied to DSS to fabricate pipe connections, flanges, forged discs, etc. The main objective of this work is thus to study the hot forging behavior of a 2205 DSS in the temperature range varying from 800 to 1250 °C. The microstructure and crystallographic texture evolutions are presented and discussed based on the expected recrystallization mechanisms that are briefly recalled. Finally, the mechanical behavior is studied through tension and nanindentation tests.

2 Material and experimental procedure

A 2205 DSS received in the form of sheets of 10 mm thick with the chemical composition given in Table 1 was used in this work. Samples of 140×30×10mm were machined and prepared for the hot forging process. This operation was conducted on LASCO machine having HK500 hammer with a forging energy of 50KJ and initial tool temperature of 200°C. The hot forging operation was conducted at temperatures ranging from 850 to 1250°C. For each forging temperature, the samples were heated for 1h, then taken separately, and forged immediately (see Fig. 1). For each temperature, forging was carried out in five passes. Due to the very high strain rate (about 1s⁻¹), the duration of each deformation step was estimated to be between 10 and 20 times shorter than the interpass time, which varied between 12 and 19 s for all processed samples and all temperatures. The characterization of the material after hot deformation was firstly done by X-ray diffraction using an INEL Equinox 1000 diffractometer equipped with a linear detector and a cobalt long fine focus X-ray source (Δ2θ = 0.03°, λ = 1.78898A, ω ≠ θ). Microstructural examination was then done using a Nikon optical microscope and a ZEISS Gemini SEM 300 scanning electron microscope equipped with EDS system. Global crystallographic texture of each phase was characterized using X-ray diffraction. Quantitative texture analysis (QTA) was performed using an INEL 4 circles diffractometer in Bragg-Brentano geometry with a cobalt point focus X-ray source (λ = 1.79024A, with 2/3 Kα1 and 1/3 Kα1 radiations). The measured pole figures (with a maximum tilt angle of 80°) are 200, 220, and 111 for the BCC phase and 111, 200, and 220 for the FCC one (Figs. 2, 3, 4, 5, and 6). The determination of the orientation distribution function (ODF) and complete pole figures were calculated using Labotex® software. Micro-texture analysis was done using EBSD attached to the SEM cited above equipped with the automatic Orientation Imaging Microscopy (OIM®) software from Tex-SEM Laboratories Inc. Scans of 400 μm x 400 μm were performed with a step of 0.5 μm. For both EBSD and X-ray techniques, texture measurements were
performed on the plane perpendicular to the normal direction (ND) of the specimens, with the initial rolling direction (RD) of the sheet taken as reference direction. For this purpose, the specimens were carefully polished with silicon carbide paper up to grade 4000, and then they were electropolished using the A2 Struers solution. From the EBSD data, the grain average misorientation (GAM)—i.e., the misorientation between each neighboring pair of points within the grain, averaged on all points belonging to one given grain—was calculated. This parameter has been recently shown to be well suited to characterize in details the recrystallized state of a hot forged Ni-based alloy [22].

The area percentage occupied by grains associated with a given GAM value was then calculated to get GAM distributions. Deformed materials are usually characterized by high GAM values due to their high dislocation density, whereas recrystallized ones exhibit much weaker GAM values due to their low dislocation density—typically below 1° [22, 23]. Based on the measurement of low GAM values (below 1°) in the as-received material associated with an annealed state (see Figs. 7 and 8), this value of 1° is indeed selected in the present work to distinguish between deformed and recrystallized states. For the present analysis, the misorientation separating grain boundaries (GB) from sub-grain boundaries (SGB) has also been set to 15°, and the percentages of low angle grain boundaries (LAGB, misorientation <15°) and high angle grain boundaries (HAGB, misorientation ≥15°) have also been evaluated in both phases.

For mechanical testing, tensile specimens were machined according to ASTM E8 standard [24]. The specimens were machined so that the tensile load direction is parallel to the initial rolling direction of the material. The tensile tests were conducted at room temperature at a strain rate of 0.5×10⁻³ s⁻¹. To quantify the local mechanical properties of each individual phase of the hot forged samples, nanoindentation

| Table 1 Chemical composition of the investigated 2205 DSS. |
|----------------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| Element | C (Wt. %) | Si (Wt. %) | Mn (Wt. %) | Ni (Wt. %) | Mo (Wt. %) | Cr (Wt. %) | P (Wt. %) | S (Wt. %) | Cu (Wt. %) | N (Wt. %) |
|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|---------|
| Sample in the furnace | Sample treated at a given temperature for 1h | Forgoing machine | Hot forged sample |

Fig. 1. The different steps of the hot forging process.
measurements were conducted using an Anton Paar NHT-3 nanoindenter with a Berkovich tip under 10 mN load and 1 mN.s\(^{-1}\) loading rate. Prior to this test, the tip was calibrated using fused silica, and the hardness (H\(_{IT}\)) and elastic modulus (E\(_{IT}\)) were calculated according to the method proposed by Oliver and Pharr [25]. To ensure the reproducibility of the obtained results, at least 15 indents were performed in each phase.

3 The expected recrystallization mechanisms and resulting textures

Before examining the microstructures, it is worth recalling briefly the expected types of recrystallization mechanisms [26, 27]. These are:

- **Dynamic recovery (DRV)**, already mentioned as a major mechanism in ferrite during hot deformation (see Section 1): the dislocations, produced continuously during strain, re-organize also continuously under the action of cross-slip and climb and tend to form well-defined structures. The percentage of LAGBs as well as the one of low GAM values increases. But neither GB migration (GBM) nor nucleation of new grains is observed at this stage. As a consequence, the crystal orientation changes are only due to plastic deformation, which can also further increase the degree of grain fragmentation in the case of significant reorientation [28]. Indeed, as soon as a dislocation microstructure is formed within a grain and some slight misorientations are created due to the presence of dislocations cell walls (or subgrain boundaries), the various dislocation cells which have slightly different orientations may behave differently under the activation of deformation glide and thus reorient slightly differently, further increasing the misorientations between cells.
- **Continuous dynamic recrystallization (CDRX)**: this mechanism is in fact quite close to DRV, and both terms are used frequently to designate the same microstructural evolution. The grains fragment continuously under the simultaneous action of dislocation cell re-organization due to recovery and reorientation due to plastic strain, which, depending on the deformation mode and initial texture of the material, may have the major effect on fragmentation. Some limited grain growth (GG) can also be observed. The texture evolution is thus mainly due to grain reorientation during plastic strain in this case, slightly altered by the fragmentation of the grains. CDRX, as DRV, has usually a major retarding effect on further recrystallization by decreasing the overall stored energy, main driving force for recrystallization processes. But this retarding effect can sometimes be compensated by a minor accelerating effect: the dislocation reorganization into cell walls may increase the mobility of some of these walls, sufficiently to allow the
transformation of a dislocation cell into a small nucleus, favoring then recrystallization.

- Discontinuous dynamic recrystallization (DDRX), usually observed in the austenite (see Section 1): in that case, nucleation and growth of new grains is observed. In most cases, DRV or CDRX is limited or completely absent beforehand, and strain provides the necessary driving force for nucleation and further growth. As nuclei are usually formed at GB, often from the bulging of one part of GB, this gives rise to so-called necklace structures. Texture evolution is then more important in that case, mainly due to the growth of the newly formed grains with different orientations that the main ones composing the deformed texture.

- Post dynamic recrystallization (PDRX), taking place during the interpass times. This type of recrystallization may occur through classical recrystallization, i.e., nucleation and growth of new grains. This is then equivalent to static recrystallization and sometimes called post-static recrystallization (PSRX). It can also occur through the growth of the grains created by DDRX; it is then called meta-dynamic recrystallization (MDRX). In that last case, no incubation time is needed then, unlike in the case of PSRX, and this process, associated with limited texture evolution, is then expected to be active during long interpass durations (compared to the deformation step duration) at high temperature, as in the present work.

For the expected textures after hot forging, as the textures of the initial material are quite pronounced, and as most of the main components characterizing the hot rolled and annealed textures are expected to be more or less stable during further compression, we can first recall the main components found in ferrite and austenite after classical rolling and annealing. It is
well known that most of the main orientations usually found after rolling and annealing in both phases of the DSS can be grouped into one single section of the Euler space namely the $\varphi_2 = 45^\circ$ $\varphi_2 = 45^\circ$ one [29]. These orientations, characterized by the classical Miller indices $\{hkl\}<uvw>$, where $\{hkl\}$ and $<uvw>$ are the indices of the rolling plane and direction respectively, are presented in Fig. 2. During hot forging, the initial textures of the two phases will be modified under the simultaneous influence of 3 main important items: (i) the possible activation of high temperature deformation systems, not documented for such high temperatures, but generally not associated with the appearance of radically new orientations [30]; (ii) the precise imposed macroscopic boundary conditions, which are not trivial to assess precisely because of the absence of lubrication; and (iii) the active recrystallization processes and especially the nucleation one which can bring new texture components. In the present case, it was not possible to separate the effects of these three items, which would need complex simulations to go a little bit further. It is therefore assumed that only discontinuous dynamic recrystallization can be responsible for significant changes in texture. It is also worth mentioning that the sole effect of annealing treatment at temperatures up to 1250 °C has already been studied in the considered material [29] and that it has been shown to be very limited, since the initial state corresponds to an annealed state. Only some very limited phase transformation, producing some limited growth of some texture components, was observed at the higher temperatures. No new component, which could have been issued from a more complex transformation mechanisms was indeed observed.

4 Results and discussion

4.1 Effect of hot forging on the microstructure and crystallographic texture evolution

Figure 3 a and b show the crystal orientation maps of both the austenite and the ferrite phases at the as-received state of the material. Both phases exhibit elongated morphologies according to the prior rolling direction of the sheet with an average grain width of about 10μm. The two phases present some morphological differences indicating that they acted differently during the prior thermomechanical history of the material (as already observed in [29, 31]). Austenite grains (Fig. 3a) contain few annealing twins and appear more fragmented than ferrite ones due to their partial recrystallized state. Ferrite grains (Fig. 3b) are still elongated in the rolling direction indicating the occurrence of only recovery during the fabrication process of the sheet, sufficient though to eliminate almost structural defects such as dislocations, as deduced from the characterization of the GAM parameter presented below. This difference in behavior of the two phases can be attributed to both the difference in the SFE and the difference in the stored energy during the deformation step that is higher after rolling in austenite than in ferrite [32, 33]. Figure 4 shows X-ray diffractograms obtained after 4 hot forging passes. The as-received state is characterized by the presence of only austenite and ferrite peaks. The patterns corresponding to hot forging at 850 °C and 950 °C contain some $\sigma$ phase peaks. This phase precipitates within the $\delta$ ferrite through the diffusion of chromium and molybdenum elements known as $\sigma$ forming
elements [34]. Table 2 displays the various microstructural parameters obtained from X-ray data analysis through a Rietveld refinement analysis conducted using the MAUD software [35]. Apart from the lattice parameters and phase percentages, this analysis allows also determining the size of the so-called coherent domains, which are the largest single crystalline elements within the grains. In the case of a deformed material presenting a dislocation microstructure within the grains, the coherent domains can be assimilated to the dislocation cells. It is observed that hot forging at 850 and 950 °C results in a significant decrease in the ferrite volume fraction as a consequence of the σ phase formation. Increasing the deformation temperature from 1050 up to 1250 °C favors the γ → δ phase transformation leading to an increase of the δ ferrite volume fraction. The coherent domain sizes for both austenite and ferrite phases are strongly modified by hot forging, compared to the as-received state. The largest sizes for ferrite and austenite were obtained after hot forging at 1150°C and 1250 °C and can be associated with very active dynamic recrystallization processes. It is also observed in Table 2 that both ferrite and austenite lattice parameters fluctuate due to internal distortions induced by the hot forging process. It should be noted that the lattice parameters calculated here for ferrite, austenite, and σ phase are consistent with those existing in the literature [36]. Additionally, the temperatures at which σ phase formation is observed during hot forging are
the same as the ones identified for \( \sigma \) phase formation during annealing after welding \([37]\).

A low magnification image of the 2205 DSS hot forged at 850 °C for four passes is presented in Fig. 5a. It reveals the presence of many cracks within the surface of the sample. The SEM micrograph presented in Fig. 5b shows the presence of precipitates at the \( \delta/\gamma \) interfaces as well as within the \( \delta \) ferrite grains, which definitely play a role in the crack initiation. The EDS analysis of these precipitates confirmed their correspondence to the well-known \( \sigma \) phase. The chemical composition of the \( \sigma \) phase obtained here (% Cr = 29, % Mo = 9.03, % Ni = 4.03, % Mn = 1.41, % Si = 0.99) is in good agreement with other results published elsewhere \([34]\). The mechanism of the \( \sigma \) phase formation, its kinetics, and its effect on the mechanical behavior of DSS were previously investigated in other papers \([31, 34]\).

Figure 6 a–i show the EBSD orientation maps obtained after hot forging at 4 different temperatures, and Table 3 gathers the quantitative data concerning the grain boundaries extracted from these maps. The common remark is that the initial elongated morphology observed in Fig. 3 is strongly affected by the hot forging process: the morphology, average grain size, degree of fragmentation, and orientations evolve indeed strongly during the whole process. This is due to both the effect of simultaneously deformation and recrystallization and the gradual evolution of the percentage of the two phases with the increase of forging temperature (see Table 2).

**Table 2.** Microstructural features extracted from the Rietveld analysis of the X-ray diffraction data

|                | As received | 850°C 4 passes | 950°C 4 passes | 1050°C 4 passes | 1150°C 4 passes | 1250°C 4 passes |
|----------------|-------------|----------------|----------------|----------------|----------------|----------------|
| **Austenite**  |             |                |                |                |                |                |
| Lattice parameter (Å) | 3.6059       | 3.6070         | 3.6205         | 3.6078         | 3.6139         | 3.6039         |
| Coherent domain size (Å) | 875.83       | 503.67         | 420            | 948.4          | 840.3          | 1288.79        |
| Phase volume fraction (%) | 48.32        | 55.30          | 49.14          | 48.7           | 42.8           | 34.9           |
| **Ferrite**    |             |                |                |                |                |                |
| Lattice parameter (Å) | 2.8829       | 2.882          | 2.8937         | 2.8803         | 2.886          | 2.878          |
| Coherent domain size (Å) | 1321.38      | 523.68         | 214            | 1257.4         | 2788           | 1497.93        |
| Phase volume fraction (%) | 51.68        | 19.79          | 24.52          | 51.3           | 57.2           | 65.1           |
| **σ phase**    |             |                |                |                |                |                |
| Lattice parameter a (Å) | //           | 8.815          | 8.8596         | //             | //             | //             |
| Lattice parameter c (Å) | //           | 4.596          | 4.617          | //             | //             | //             |
| Coherent domain size (Å) | //           | 358.805        | //             | //             | //             | //             |
| Phase volume fraction (%) | //           | 24.92          | 26.34          | //             | //             | //             |
The morphology and microstructural characteristics of the ferrite and austenite phases present at 850°C (Fig. 6a–b) differ from those obtained at higher temperatures (above 1050°C). This is due to two reasons: (a) the 1-h pre-heating time before hot forging leads to some grain growth and morphological change, mainly in austenite, since no transformation occurred in this phase at that temperature, and (b) the presence of hard σ phase particles that affects the deformation of the softer austenite and ferrite phases during hot forging. This contributes to the strong increase of the LAGBs percentage and subsequent drop of the HAGBs one in that case. Due to the relatively small amount of σ phase and its more brittle character, the GB percentages are less significant in this phase.

A large re-increase of the HAGBs percentage in both austenite and ferrite phases is observed at 1050 °C, compared to 850°C. In the austenite phase (Fig. 6c), the grains undergo significant fragmentation and evolve towards a cellular morphology. This is accompanied by slight changes of the orientation colors, indicating that the processes of DDRX and PSRX did not take place. It can thus be assumed that only CDRX and/or MDRX have occurred in this case. On the other side, the rapid diffusion kinetics within ferrite and its high stacking fault energy compared to austenite facilitates the dislocation rearrangement and annihilation. This favors in turn the modification of the sub-boundaries formed during straining and leads to the formation of polygonal ferrite grains as shown in Fig. 6d. Thus, DRV or CDRX and MDRX can be adopted as softening mechanisms in the ferrite phase during hot forging. This is accompanied by an important texture evolution resulting from grain reorientation during straining; the plane strain compression may reinforce the {111} and {100} components in bcc materials.

Increasing the deformation temperature to 1150°C produces further significant microstructural changes. The EBSD orientation map (Fig. 6e) shows that austenite evolves towards a cellular morphology after 4 passes hot forging at this temperature. Its grains contain some HAGBs that are not close to become new grain boundaries. This suggests that the CDRX process, followed by MDRX, is not complete. At the same time, the ferrite microstructure undergoes a remarkable change; the percentage of LAGBs is strongly reduced from 78% at 1050°C to 23% at 1150°C. Apparent grain growth is also observed (Fig. 6f). This suggests the activation of either DDRX and/or PSRX in this case. A further increase of the deformation temperature up to 1250 °C results on the one hand, in the austenite and ferrite grain growth and, on the other hand, in the enhancement of the γ → δ phase transformation (the EBSD maps are not presented here for reasons of paper conciseness). As indicated in Table 2, the austenite volume fraction decreases from 48.7 (at 1050 °C) to 34.9 % (at 1250°C) due to the γ → δ phase transformation that occurs through a grain boundary migration process. In the austenite phase, the mechanism of CDRX, followed by MDRX, seems to be the main one, whereas in ferrite, the increase of the LAGBs percentage suggests that at 1250°C, we do have CDRX possibly after DDRX as at 1150°C. A strong correlation between the coherent domain sizes (d) measured by X-ray diffraction (Table 2) and the percentage of LAGBs for the ferrite phase can be observed (Table 3). Indeed, the more the LAGBS are formed, the smaller is the size of the dislocation cells, represented by that of the coherent domains. This indicates that the process of grain fragmentation during hot forging depends, in a complex way, on the initial state of the material and possible recrystallization mechanisms. Figures 7 and 8 show the GAM distribution for austenite and ferrite in the investigated DSS. Both ferrite and austenite at the as-received state exhibit a prominent GAM peak below 1°, indicating a total annealed state in both phases. Table 3 shows that ferrite and austenite phases have similar average GAM values equal to 0.79 and 0.76, respectively, even though the principal annealing mechanisms are different in both phases. After hot forging, the GAM distribution evolves differently in the two phases. In the austenite phase (Fig. 7), a significant dispersion is observed in the GAM distribution for all temperatures, although it is slightly reduced at the highest temperature.

The fraction of recrystallized grains is very small (below 1%) at 1050 and 1150°C (see Table 3). In the ferrite phase...
Fig. 7 GAM histograms assessed for the austenite phase at the as-received state and after hot forging at various temperatures. The black dashed line indicates the difference between recrystallized from deformed grains.

Fig. 8 GAM histograms assessed for the ferrite phase at the as-received state and after hot forging at various temperatures. The black dashed line indicates the difference between recrystallized from deformed grains.
(Fig. 8), we can say first that all samples comprise a percentage of recrystallized grains (Table 3) and that the GAM distribution of ferrite presents always less dispersion than that of austenite: especially, the maximum value is always lower in the ferrite phase. Also, it is quite clear that the ferrite is completely recrystallized after forging at 1150°C. These observations are in good agreement with the mechanisms proposed above: CDRX, very active in the austenite phase, produces an increase of the average GAM, whereas DDRX, active in the ferrite phase at 1150°C, produces a drastic decrease of the average GAM. Also, the fact that the average GAM is the highest in the ferrite phase deformed at 1050°C is in favor of the sole CDRX (or DRV) process at this temperature. It can thus be concluded that, up to 1050°C, the reduction of stored energy due to DRV is sufficient to retard DDRX, and thus only CDRX is visible at this temperature, whereas at 1150°C, while the stored energy decreases due to DRV, the mobility of some dislocation cells becomes high enough to produce DDRX. Above 1150°C, this effect, although maybe present, is annihilated by the further decrease of SFE, and again CDRX becomes predominant at 1250°C.

As for the possible occurrence of PDRX mechanism, we can say that, because of the short deformation times and long interpass times, MDRX is most probably active in all cases, but not PSRX, which needs an incubation time to start, since it would have led to much larger proportions of recrystallized grains and a drastic reduction of the dispersion in the GAM distribution.

It is now important to examine the effect of the hot forging process on the crystallographic texture evolution of the studied DSS. The \(\varphi_2 = 45°\) ODF section of the as-received state (Fig. 9a) indicates that austenite crystallographic texture is mainly composed of a major Brass=\{110\}\{112\} (Bs) component with a maximal intensity of 5.5. A weak Goss=\{110\}\{001\} component is also observed. These texture components have been typically observed in austenite in other research works after rolling and annealing [38, 39]. The crystallographic texture of the ferrite phase in the as-received state (Fig. 9b) is mainly composed of the rotated cube = \{001\}\{110\} component, with some spread along the \{001\} //ND fiber. It is interesting to note that there exists a marked orientation relationship typical from the \(\delta \rightarrow \gamma\) phase transformation, between the main component of each phase. the misorientation between the rotated cube orientation in the ferrite phase and the Bs orientation in the austenite phase is indeed equal to 45.99° around a \([0.2, 0., 0.976]\) axis, which is very close to the Nishiyama-Wasserman relationship [40, 41]. It is also worth noting that, based on the assumption of the main activation of low temperature slip systems (i.e., \{110\}<111> and \{112\}<111> within the \(\alpha\) phase and \{111\}<110> within the \(\gamma\) phase), the \{111\}<uvw> and \{100\}<uvw> components are expected to be stable during forging or uniaxial compression within the \(\alpha\) phase, whereas the \{110\}<uvw> components are expected to be stable within the \(\gamma\) phase. Additionally, because of the axi-symmetry of the forging process, the increase of the fiber-like character of the textures, with a fiber axis parallel to the compression direction, is also expected. The main expected fibers are plotted in Figs. 9, 10, and 11 as dotted red lines.

During hot forging (Figs. 10 and 11), there is a general spreading of the texture components along \{hkl\} fibers (dotted lines) in both phases. This can be attributed to the effect of uniaxial compression and the grain fragmentation described above. In the austenite phase, a strong weakening of its crystallographic texture after hot forging at 850° is observed (Fig. 10a). This spreading is consistent with the fragmentation and
reorientation of the grains during forging. Hot forging at 1050°C leads then to the appearance of the rotated Goss={110}〈110〉 component (Fig. 10b) in addition to the existing weak Bs component. The rotated Goss component is further reduced at 1150°C (Fig. 10c), while new components close to {113}<110> appear. Increasing the deformation temperature to 1250°C produces a strong change of austenite crystallographic texture (Fig 10d): both rotated Goss and Bs components have disappeared, and a {111}〈110〉 texture component is now observed in the austenite phase, which is quite unusual in this phase.

In the ferrite phase, the rotated cube component disappears during hot forging at 850°C and is replaced by a weak texture distributed along several fibers (Fig. 11a). Increasing the forging temperature from 850 to 1250°C (Fig. 11b to d) further modifies the crystallographic texture of the material, which becomes composed of a main {001} // ND fiber at 1150°C and a main {113}// ND fiber at 1250°C (Fig. 11d). It should be noticed that the texture found within the ferrite phase at 1250°C is quite unusual, although {113} components are often observed in electrical ferritic steels after recrystallization [42]. This texture is the result of a complex thermomechanical path for which some important details are not known, such as the possible active deformation systems at high temperature, the exact deformation path, or the degree of recrystallization. In any case, the quite unusual texture components observed at the highest forging temperature could be due to the simultaneous effect of partial DDRX within the ferrite phase, which produces a {113} fiber, and to the presence of an additional “in-plane” shear component (like in asymmetrical rolling).
which, in the extreme case, tend to “invert” the BCC and FCC rolling textures. This additional shear may explain the presence of some unusual texture components at the highest temperature, and especially the presence of a \{111\} fiber in the \(\gamma\) phase and of a \{112\} fiber in the \(\alpha\) phase [43, 44]. Unlike in the case of ferrite at 1150 °C, these components cannot be attributed to DDRX since they are not associated with a drastic reduction of the GAM values.

### 4.2 Investigation of the mechanical behavior

The true stress-true strain curves of the 2205 DSS hot forged at different temperatures are given in Fig. 12. The mechanical properties determined from this figure for each deformation domain are given in Table 4. Both yield strength \(\sigma_y\) and tensile strength \(\sigma_u\) increase significantly after hot forging at 850°C in comparison to the as-received state and then decrease continuously with a further increase of the forging temperature up to 1250°C. At the same time, the fracture stress \(\sigma_f\) drastically decreases after hot forging at 850°C; it increases again for higher forging temperatures but remains always lower than in the as-received state. The yield strain \(\varepsilon_y\) is hardly modified by the hot forging process whatever the temperature. Both maximum strain \(\varepsilon_u\) and fracture strain \(\varepsilon_f\) decrease drastically after hot forging at 850°C in comparison to the as-received state. As mentioned previously, the \(\sigma\) phase precipitation at this temperature limits the dislocation movement at the vicinity of the grain boundaries. The \(\sigma\) phase particles destroy the microstructural continuity at the \(\delta/\gamma\) interfaces [45] and form an inescapable barrier that prevents the dislocation mobility at these interfaces during deformation. Hence, the dislocations ensuring the plastic straining of austenite and ferrite are

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Fig. 11 ODF (\(\varphi_2 = 45^\circ\)sections) of ferrite after 4 passes hot forging at a 850°C, b 1050°C, c 1150°C, and d 1250°C.
blocked at the γ/σ and δ/σ interfaces and resist to the plastic deformation process. Consequently, the material’s damage increases at these interfaces due to the increase of the stress and strain levels and the presence of hard particles that resulted in the drastic loss of ductility (Table 4) and the cracking phenomenon illustrated before in Fig. 3a. The occurrence of the plastic flow after hot forging at 850 and 950 °C requires higher strength values that resulted in the increase of the material’s yield strength. Hot forging at temperatures ranging from 1050 to 1250 °C resulted in improved mechanical properties compared to those obtained at 850 and 950°C due to, on the one hand, the absence of the σ phase and, on the other hand, to dynamic recovery and recrystallization occurring at these temperatures. The best compromise between ductility and resistance to plastic deformation is evaluated through the calculated values of the product σ_u·ε_u (Table 4). In that sense, once the negative influence of the σ phase is suppressed, all 3 higher temperatures are more or less equivalent, since this parameter varies between 2.1 and 2.2.

Nanoindentation measurements conducted in ferrite, austenite, and σ phase are displayed by the typical load-displacement (P-h) curves shown in Fig. 13a. The data collected from Fig. 13a shows that the elastic modulus (E_IT) values of austenite are slightly higher than those of ferrite (335 ± 4 GPa and 330 ± 2 GPa, respectively), while the nanohardness (H_IT) of ferrite is lower. This is in good agreement with the results found by Kheradmand et al. [46]. The difference in microscopic residual stresses between ferrite and austenite grains, with different thermal expansion coefficients, allows the nitrogen element to play a crucial role in stabilizing austenite and acts as a planar slip promoter that enhances the strength of austenite [47–49]. As expected, the indentation depth of σ phase (red line in Fig. 13a) is less deep than that of austenite and ferrite phases. This indicates that the nanohardness and Young’s modulus of σ phase are the

| Forging temperature | As received | 850°C | 950°C | 1050°C | 1150°C | 1250°C |
|---------------------|------------|-------|-------|--------|--------|--------|
| Yield strength σ_y  | 495.82     | 807.48| 766.17| 687.68 | 604.91 | 593.86 |
| Yield strain ε_y in %| 1.34       | 1.76  | 1.83  | 1.63   | 1.79   | 1.61   |
| Tensile strength σ_u| 1076.7     | 1173.7| 1154.4| 1133.9 | 1059.3 | 994.31 |
| Maximum strain ε_u in %| 31.98 | 3.73  | 11.41 | 19     | 20     | 22.09  |
| Empirical parameter σ_u·ε_u| 3.4  | 0.44  | 1.3   | 2.15   | 2.11   | 2.2    |
| Fracture stress σ_f | 643.22     | 150.83| 620.73| 781.34 | 641.18 | 574.49 |
| Fracture strain ε_f | 0.3864     | 0.0814| 0.1495| 0.2583 | 0.2805 | 0.2974 |
highest (8.2 ± 0.6 GPa and 342 ± 2 GPa, respectively) because of the elements nature (Cr-Mo), as well as the complexity of its crystalline structure [50].

Figure 13 b shows P-h curves of ferrite for samples subjected to 4 passes hot forging at different temperatures. The HIT and EIT values, corresponding to the as-received state, are 3.2 ± 0.3 GPa and 274 ± 4 GPa, respectively. Hot forging at 850°C increased the hardness and the Young’s modulus of the steel (7.4 ± 0.2 GPa and 329 ± 3 GPa, respectively). A further increase in forging temperature up to 1250 °C resulted in a decrease of these characteristics. The observed increase in the mechanical properties is attributed to the work hardening that was induced by the dislocation nucleation phenomena [51]. In general, after the plastic deformation, in that case hot forging, the change of bonding force between atoms causes a decrease in elastic modulus. The main reasons to that are impurity, secondary phases, grain orientations, dislocations, and so on [52]. In the present work, the high elastic modulus of σ phase can explain the increase in elastic modulus after 4 passes hot forging at 850°C and 950°C. However, after hot forging at high temperatures (1050–1250°C), the important texture evolution observed in ferrite caused by the CRDX discussed in Section 4.1 resulted in a softened microstructure due to the low dislocation density with relatively low HIT and elastic modulus.

A zoom view of loading segment of P-h curves (Fig. 13b) shown in Fig. 14a–c exhibits a significant difference during loading process of the as-received, 850°C and 1250°C 4 passes hot forged samples. A distinguished sudden displacement, known as pop-in, is observed in the initial part of P-h curves in the as-received sample and barley detected after 4 passes hot forged at 850°C sample. This phenomenon disappears for further hot forging temperatures. This is attributed to the elastic straining by nanoindentation test that can be fitted using the Hertzian elastic contact solution according to Eq. (1), where the tip shape is considered spherical at shallow depths [53] as illustrated in Fig. 14 a and b.

\[
P = \frac{4}{3} E_r \sqrt{R h} \frac{h}{3}^{1/2}
\]

where P is the applied load, \( E_r \) is the reduced Young’s modulus, \( R_i \) is the curvature of the indenter (500 nm), and h is the displacement into surface.

It was assumed that the first pop-in (Fig. 14a) is generally related to the drastic homogeneous dislocation nucleation or dislocation source activation [51] where the ferrite phase starts to deform plastically in the very early stage of the indentation process (250–300 μN). Taking into account the large tip radius and irregularity in tip geometry, the critical shear stress value for the homogeneous dislocation nucleation cannot be presented with absolute precision. Nevertheless, the maximum shear stress (\( \tau_{max} \)) underneath the indenter calculated in the as-received condition using Eq. (2) is found to be (4.2 GPa) in the same order of the theoretical strength of the free-defect iron crystal \( (\frac{\tau}{15} < \tau < \frac{1}{15}) \) where the shear modulus of ferrite is approximately 83 GPa [53]. This suggests that the pop-in is occurred in free-defect area.

\[
\tau_{max} = 0.18 \left[ \frac{6 \pi}{\pi^3} \frac{P E_r^2}{R^2} \right]^{1/3}
\]

Hot forging at 850°C resulted in the lack of pronounced pop-in effect (Fig. 14b) that is explained by the movement and multiplication of unlocked existent dislocations by high shear stress. It is clear that the dislocation density in the deformed material play a key role in the pop-in occurrence. The high dislocation density exists under the indenter tip in the pre-strained material, i.e., 4 passes hot forged sample at 1250°C, which promotes the dislocation activation and multiplication under a lower shear stress rather than the nucleation of new dislocation at very high stresses [54, 55]. Consequently, the disappearance of pop-in indicates that elastic-plastic...
deformation occur right at the beginning of the indentation experiment in highly deformed material (Fig. 14c). Therefore, based on nanoindentation tests, hot forging at 850°C and 950°C induced work hardening of ferrite phase by dislocation nucleation phenomena. The presence of $\sigma$ phase (with high $H_{IT}$ and $E_{IT}$) at these temperatures is the origin of the high macro-mechanical properties given in Table 4. On the other side, the softening microstructure of ferrite obtained after hot forging at high temperature (1050–1250°C) as a result of CRDX, caused a decrease in hardness and elastic modulus that comes down to reduce the global mechanical properties already issued from uniaxial tensile test.

5 Conclusions

The main conclusions of the conducted microstructural and mechanical investigations are given as follows:

- Hot forging of the 2205 DSS at temperatures below 950°C resulted in a sharp cracking phenomenon due to the combined effect of both $\sigma$ phase precipitation and severe plastic strain that affected drastically its mechanical properties.

- Distinguished recrystallization mechanisms took place during hot forging depending on the processing temperature. In the ferrite phase, DRV or CDRX followed by MDRX was found to be principally active up to 1150°C. It was then massively supplemented by DDRX and PSRX at 1150 °C, presumably because of a favorable compromise of SFE reduction and enhanced migration rate at this temperature, which was again supplemented by massive CDRX and MDRX at 1250 °C. The $\{113\}$ component which appears at 1150°C and 1250°C could be attributed to DDRX. In the austenite phase, CDRX was observed for all forging temperatures.

- The different forging temperatures resulted in a significant texture evolution that was attributed to the simultaneous action of grain reorientation due to plastic strain, recrystallization and transformation processes. The final textures presented a fiber-like character, mainly due to the uniaxial character of the compression process.

- The observed crystallographic textures after forging at 1250°C in both phases were found to be quite unusual. A strong additional in plane shear component due to friction effects, supplemented by partial DDRX, could be responsible for this evolution.

- The forging temperature variation strongly affected the mechanical behavior of the investigated material. While
a sharp increase of strength and drastic loss of plasticity were recorded after hot forging at 850°C, a progressive gain of plasticity is noticed when increasing the forging temperature from 1050 to 1250°C.

- The microstructural and mechanical investigations conducted in this work allow us to conclude that the best combination of microstructure and mechanical properties of the hot forged 2205 DSS was obtained after hot forging at 1050°C, which precisely corresponds to the temperature range for which CDRX and MDRX are massively active in both phases.

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Availability of data and material The raw/processed data required to reproduce these findings cannot be shared at this time as they will be used in an ongoing study.

Code availability Not applicable (jmp 13 design of experiments “free version”)

Declarations

Ethics approval This study complies with the ethical standards set out by Springer. All the authors read and approved the final manuscript.

Conflict of interest The authors declare no competing interests.

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