A study of the static recrystallization behaviour of cast Alloy 825 after hot-compressions

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Abstract. The static recrystallization behaviour of a columnar and equiaxed Alloy 825 material was studied on a Gleeble-3800 thermo-simulator by single-hit compression experiments. Deformation temperatures of 1000-1200 °C, a strain of up to 0.8, a strain rate of 1s⁻¹, and relaxation times of 30, 180, and 300 s were selected as the deformation conditions to investigate the effects of the deformation parameters on the SRX behaviour. Furthermore, the influences of the initial grain structures on the SRX behaviors were studied. The microstructural evolution was studied using optical microscopy and EBSD. The EBSD measurements showed a relaxation time of 95 % for fractional recrystallization grains, t₉₅, in both structures, was less than 30 seconds at the deformation temperatures 1100 °C and 1200 °C. However, fewer than 95% of recrystallized grains recrystallized when the deformation temperature was lowered to 1000 °C. From the grain-boundary misorientation distribution in statically recrystallized samples, the fraction of high-angle grain boundaries decreased with an increasing deformation temperature from 1000 °C to 1200 °C for a given relaxation time. This was attributed to grain coarsening.

1. Introduction
The Nickel-based alloy 825 is resistant to many corrosive environments [1-3]. Moreover, alloying it with small amounts of carbon provides strengthening. As shown in previous reports [4-5], dynamic recrystallization in either the columnar or the equiaxed compressed structure did not occur at any hot-working temperature. Industrial products, particularly billets or bars, manufactured from the alloy are subjected to high deformation temperatures and exposed deformation schedules which are varied to meet property requirements. The combined effects of the deformation temperature and relaxation time on the material lead to microstructural changes, which significantly affect the mechanical properties. In practice these microstructural changes can include recovery and static recrystallization. In literature, it has been shown that the deformation temperature and holding time have a large influence on the softening or recrystallization of the material structure [6-7]. The static-recrystallization kinetics in columnar and equiaxed grain structure have been mapped by using hot compression testing. The fraction recrystallized structure was evaluated by an EBSD study of rapidly quenched specimens. This method is based on the observation of microstructural evolution with relaxation time. This paper will focus on understanding the static recrystallization behaviour over a range of deformation temperatures (1000 °C, 1100 °C, and 1200 °C), relaxation time (30 sec, 180 sec, and 300 sec), at a constant strain rate of 1s⁻¹ and a strain of 0.8, respectively.
2. Experimental material

The investigated alloy was prepared via melting in the Electric Arc Furnace, refining on the Argon Oxygen Decarburisation converter and then Continuous Casting of blooms. The chemical composition of this investigated heat is shown in Table 1.

|   | C   | Si  | Mn  | Cr  | Fe  | Mo  | Ti  | Cu  | N   | Ni  |
|---|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
|   | 0.007 | 0.23 | 0.67 | 22.1 | 32.0 | 2.53 | 0.8  | 1.6  | 0.009 | Bal. |

The cylindrical compression samples of a 15 mm height and a 10 mm diameter were cut and machined with the compression axis parallel to the continuous cast axis from a constant radial position to the columnar and equiaxed grain zones. To study the softening behaviour of Alloy 825 at different temperatures, monotonic-hit compression was employed using a Gleeble 3500 Thermo-Mechanical Simulator. The deformation temperature tests were carried out at a deformation temperature of 1000 °C, 1100 °C and 1200 °C with a nominal strain of 0.8 and a constant strain rate of 1 s⁻¹. The deformed samples were held at the test temperature for relaxation times of 30, 180, and 300 seconds before undergoing the fast cooling. For more details about experimental procedure and similar flow stress curves the reader is referred to reference [5]. The percentage static recrystallized (SRX) grain and microstructural evolution at different testing conditions were studied by using electron backscatter diffraction and optical microscopy.

3. Results and discussion

3.1. Effect of deformation conditions on microstructure

Figure 1 shows an example of the microstructure of relaxed columnar and equiaxed structures of Alloy 825 samples, after the hot compression with different deformation conditions. A partial recrystallization occurred in both relaxed structures as shown in the micrographs, figure 1 (a and b), and recrystallized grains forms on grain boundaries and at large particles (as shown with PSN). This is shown by the presence of some grains that are much smaller than others, as shown by arrows. Additionally, static recrystallized grains observed in the equiaxed structure was larger than in the columnar structure and the fraction of static recrystallized grains in the equiaxed structure was larger than in the columnar structure.

![Figure 1](image1.png)

**Figure 1.** Optical microstructures at the center area of the columnar (C) and equiaxed (E) samples after completion of the SRX tests at the indicated deformation temperature and relaxation time.
After a relaxation time of 300 s, the volume fraction of statically-recrystallized grains increased in both the relaxed columnar and equiaxed samples, figure 1(c and d). However, SRX grains became progressively much coarser in both the columnar and equiaxed specimens. A recovery and an almost full recrystallization occurred, as shown in the micrographs, figure 2e. Also, a small amount of static recrystallized grains was already coarsened. Additionally, statically-recrystallized grains observed in the columnar structure were coarser than in the equiaxed structure. A full recrystallization occurred as shown in the micrograph, figure 1f, and a large amount of static recrystallized grains were already coarsened. Additionally, statically-recrystallized grains observed in the columnar and equiaxed structure was similarly coarsened.

3.2. Hardness of deformed columnar/equiaxed Alloy 825

Table 2 shows variation of the Vickers microhardness with the relaxation time at given deformation temperatures of 1000 °C, 1100 °C, and 1200 °C in columnar and equiaxed relaxed samples, together with the standard deviations. The columnar and equiaxed recrystallized samples showed that the hardness level decreases with an increasing deformation temperature and relaxation time, reaching a lowest value of hardness indicative of softening via recovery.

Table 2. Hardness (HV₀.3) of recrystallized columnar/equiaxed Alloy 825, with ± standard deviation.

| Structure      | Deformation temperature, [°C]/Relaxation time, [sec] |
|----------------|-----------------------------------------------------|
|                | 30  | 180 | 300 | 30  | 180 | 300 | 30  | 180 | 300 |
| Columnar       | 160±5 | 157±7 | 154±4 | 162±4 | 141±7 | 143±6 | 138±2 | 131±7 | 131±11 |
| Equiaxed       | 175±6 | 151±12 | 150±4 | 147±11 | 147±8 | 146±7 | 138±12 | 136±8 | 134±1 |

3.3. Grain misorientation distribution of relaxed samples

The recrystallized grain distribution of misorientation angles for columnar and equiaxed alloy 825 deformed microstructures, for a 5° grain definition, is shown in figure 2. Figure 3 supports the conclusions drawn from figure 2 (a and b) at a more quantitative level. In the columnar regions, figure 2a showed that the frequency of LAGB was somewhat higher than the frequency of HAGB, due to onset of the limited amount of dynamic subgrains recrystallization (DRX subgrains). At a relaxation time of 30 s, the frequency of HAGB increases with increasing deformation temperatures due to the SRX grains increases in size and a type of grain-growth occurs. However, the frequency of HAGB obtained at a deformation temperature of 1200 °C was approximately 75% lower than that obtained at 1100 °C due to grain coarsening. Beyond a relaxation time of 30 s, the amount of HAGB diminishes as the deformation temperature increases. This is indicative of increasing grain sizes as grain growth occurs and grains become coarser. This supports the conclusions drawn from Table 2 and figure 4b. In the equiaxed condition and at a given relaxation time, the decreasing fraction of HAGB with increasing deformation temperatures is due to grain coarsening. However, approximately 70% of boundaries detected in the equiaxed structure at a relaxation time of 180 s, figure 2c, were high angle boundaries (60°) and the remainder were low angle boundaries (<5°). This is due to low temperature, which leads to a mixture of both refined and coarse grains.

3.4. Percentage static recrystallized grains and grain size, d_{SRX}

The plot of measurements of the recrystallization percentage versus relaxation times in columnar and equiaxed compressed samples in the temperature range of 1000 °C and 1200 °C is shown in figure 3a. The fractional recrystallized grains of columnar and equiaxed specimens deformed to various deformation temperatures and relaxed at different relaxation times revealed that the fraction of SRX recrystallized grains is higher than 80%. As with the tests performed at 1000 °C, a clear sigmoidal shaped curve is shown for the columnar and equiaxed specimens with incomplete static recrystallization occurring until a relaxation time a relaxation time of 300 s, where more than 80% recrystallization was observed. It could be noted that the SRX recrystallization curve for the lowest deformation temperature
1000 °C, showed sigmoidal behaviour with the rate of SRX recrystallization decreasing after a relaxation time of 180 s. The fraction of SRX grains increases sharply at the intermediate deformation temperature 1100 °C up to a relaxation time of 30 s, where more than 80% and 90% recrystallization degrees in columnar and equiaxed, respectively. Thereafter, this becomes constant beyond 30 s which signifies to a fact that the relaxation times have no significant effect on SRX fraction. However, fractions of SRX grains are 88-90% for both columnar and equiaxed structures and more than 95% recrystallization was observed after 30 s. At deformation temperatures of 1100 °C and 1200 °C, full SRX recrystallization occurred within 180 s for both structures. Measurements of the mean recrystallized grain size versus deformation temperatures in columnar and equiaxed deformed samples at relaxation times of 30 s, 180 s, and 300 s compared to un-relaxed samples (the values obtained from our previous study [5]) are shown in figure 3b. As is shown, the average recrystallized grain size generally increases when increasing the deformation temperature at a given relaxation time. In both the columnar and equiaxed deformed samples, the average SRX grain size sharply increases with an increased relaxation times from 0 s to 30 s. It is obvious that the average SRX grain size in columnar deformed specimens for relaxation time 0 s was 15% lower than of equiaxed deformed samples. In columnar deformed specimens, the average recrystallized grain size is observed to be similar for relaxation times of 180 s and 300 s. It is apparent that the relationship between the average SRX grain size and the deformation temperature in both columnar and equiaxed specimens is almost linear.

3.5. The inverse pole figure (IPF) maps

Figure 4 shows a few of examples of the EBSD orientation mapping images of the relaxed specimens for relaxation times between 30 s and 300 s over a range of deformation temperatures of 1000 °C, 1100 °C, and 1200 °C and at a constant strain rate of 1 s⁻¹ and a strain up to 0.8. The color of EBSD grains refers to the different orientation and the similar color shows that the grains have a similar orientation, as can be seen in the IPF coloring in figure 4g. The texture for both structures is a double fiber texture with <110> and <100> parallel to the compression direction. This is likely dominated by the large
non-recrystallized grains, as shown in figure 4 (a and b). For both structures, subgrain structures form low angle grain boundaries are predominated during hot deformation at a temperature of 1000 °C with a relaxation time of 30 s (recrystallization by subgrain rotation may occur.[7]). At the 30 s relaxation time, large amounts of coarse and deformed grains (non-recrystallized) together with fine recrystallized grains are formed within both structures, (rotation mechanism, [7]), figure 4 (a and b). In the equiaxed structure, the SRX occurs through the so-called necklace mechanism, forming at some individual deformed grain boundaries, figure 4b. At a prolonged relaxation time of 300s, there were more statically recrystallized grains than after holding for 30s and the almost no deformed substructure was visible in the recrystallized microstructure, figure 4 (c-d). Furthermore, the SRX grains and size increased and a small amount of the non-recrystallized grains still could be found in the microstructure. At a temperature of 1100 °C and a relaxation time of 30 s, the large deformed grains almost disappear and the refinement of SRX grains and twins can predominately be seen in the microstructure, figure 4 (e). Large SRX grains (grain growth) can be observed and annealing twins can still be identified in the whole columnar microstructure, figure 5e. As shown in figure 4 (e and f), the number of statically-recrystallized grains decreases when increasing the deformation temperature at a given relaxation time because of grain growth. At a deformation temperature of 1100 °C, in a columnar relaxed specimen, the number of recrystallized grains significantly increases beyond a relaxation time 30 s. This is due to the nucleation of more SRX grains (figure 3a supported this) and then gradually decreases when reaching a relaxation time of 300s due to grain coarsening. The latter, the lowest deformation temperature combined with a long relaxation time, could lead to either the nucleation of more SRX grains or a growing of smaller SRX grains. Nevertheless, in equiaxed relaxed specimen, the amount of recrystallized grains slightly decreases from a relaxation time of 30s to 300s, which indicates that grain coarsening occurs. As previously discussed, the effect of increasing the relaxation time corresponds to an increase of the average recrystallized grain size and percentage of recrystallized fraction. It is obvious that the deformation temperature, below or at a relaxation time of 30 s, has a strong influence on the onset of SRX fraction and average recrystallized grain size in both columnar and equiaxed specimens. The relaxation time, after 30 s at the lowest deformation temperature of 1000 °C or before that time at the intermediate and highest temperature has only a slight effect on the SRX fraction at the later stages and/or on the average recrystallized grain sizes in both columnar and equiaxed specimens.

![Figure 4](image-url). Microstructural evolution of the relaxed columnar (C) and equiaxed (E) specimens at the indicated deformation temperatures and relaxation times. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this paper.)
4. Conclusions
The SRX behaviour in a columnar and equiaxed deformed structure of an Alloy 825 was studied in the temperatures range of 1000 to 1200 °C with different relaxation times: 30 s, 180 s, and 300 s. Tests were carried out using at a constant strain rate of 1 s\(^{-1}\) and a nominal strain of up to 0.8 through single-hit compression tests using a thermomechanical simulator. The microstructural evolution was studied using LOM and EBSD. The primary outcomes of the study are as follows:

1) The time of a 95% recrystallization, \(t_{95}\), in both samples commences the time for a complete recrystallization, was less than 30 s at the intermediate and highest deformation temperatures of 1100 °C and 1200 °C. However, a 95% recrystallization was not achieved at the lowest deformation temperature (1000 °C) for any relaxation time.
2) The average SRX grain size, in columnar/ equiaxed grain samples, increases 3-5% with increasing deformation temperatures. Beyond a 30 s relaxation time, the grain size increases almost linearly with an increased deformation temperature.
3) In general, the fraction of SRX and the recrystallized grain size increased with an increased deformation temperature combined with an increased relaxation time. At the lowest deformation temperature of 1000 °C, the relationship between the fraction of SRX and the relaxation time is a typical sigmoidal shape.
4) From grain-boundary misorientation distributions in both statically recrystallized structures shows that the decreasing amount of HAGB decreased with an increased deformation temperature at a given relaxation time, due to a grain coarsening.

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