The possibility of full control of the grain size, and hence the production of products with a fine grain structure are demonstrated by HSLA (High Strength Low Alloy) containing micro-additives with high affinity with carbon and nitrogen, such as Nb, Ti and V. The metallic micro-additives added to these steels interact with C and N to form stable interstitial phases MX (M - Nb, Ti, V; X - N, C). The stability of these phases depends mainly on their chemical composition, while the temperature of their dissolution can vary widely. In the technological process of producing steel products, this allows for obtaining various structural effects, such as: controlling the austenite grain size at high temperature by undissolved particles of precipitates, changing the kinetics of recrystallization and phase changes caused by dissolved elements and precipitation, as well as obtaining the effect of strengthening by low dispersive discharge temperature. Dispersive particles of interstitial phases type MX, such as carbides, nitrides and carbonitrides, introduced into the micro-additives steel, inhibiting the movement of grain boundaries of recrystallized austenite, create the possibility of producing metallurgical products with high mechanical properties [30-33]. The condition for obtaining metallurgical products with high mechanical properties from micro-alloy steel is the proper selection of plastic forming conditions, adapted to the separation process of MX type interstitial phases [15, 37]. In microalloyed steels, the effect of MX-type phase particles on dynamic recrystallization depends on their size, shape and distribution in the matrix. If the MX-type phase particles are fine and of high dispersion, they stabilize substructure, hinder the formation of recrystallization
fronts and their migration and inhibit dynamic recovery. As a result, the impact of such particles inhibits dynamic recrystallization and may result in significant grain refining. In contrast, when the second phase particles are precipitated within the grain boundaries, they can effectively block their migration, that is inhibit dynamic recrystallization without significantly affecting dynamic recovery. For large second phase particles that interact with dislocations as stress concentrators and are also privileged points for heterogeneous formation of recrystallization nuclei, the acceleration of dynamic recrystallization is observed [20-23, 35].

Hot deformation parameters have a decisive impact on the formation of secondary phases of microadditions that determine the course of dynamic recrystallization and structure refinement in HSLA steels [1, 2, 39]. Many strategies have been adopted to improve the ductility and impact properties of these ultrahigh-strength DP (Dual Phase) steels, such as refining of microstructure, alloying, or designing new processing methods [6, 19, 28]. HSLA steel has better mechanical properties, including high strength, and better corrosion resistance than carbon steel [4, 11, 12, 45]. Mechanical properties are usually determined by chemical composition and thermo mechanical treatments [10, 13, 25, 27, 36, 38, 44]. Nevertheless, final mechanical properties are observed to depend on the fraction of reverted austenite, which is very sensitive to heat treatment [3, 8, 16, 24, 29, 41].

Meeting the growing requirements of steel customers related to the increase in strength while maintaining the required ductility is seen in the full use of micro-additives. In the case of HSLA micro-alloy steels, the increase in strength is associated with grain grinding and precipitation hardening by Nb, Ti and V carbide, nitride and carbonitride nitrides. The analysis of the literature indicates that the conducted research in a significant number of cases focus on the introduction of micro-additive V [14, 17, 26, 34, 40] and Ti and V [7, 9, 18, 42, 43]. Much less work concerns the overall introduction to steel to Nb, Ti and V steel. The purpose of this work is to investigate the impact of temperature and strain rate on the size of austenite grains of dynamically recrystallized HSLA steel with Nb, Ti and V micro-additives.

### MATERIALS AND METHODS

The investigations were carried out on the HSLA steel grade S355NL from the industrial process with the chemical composition as presented in Table 1.

The plastometric investigations with the axisymmetric hot compression method were carried out for HSLA steel to determine the effect of the austenitizing temperature and the strain temperature, as well as the degree and rate of the $\sigma$-$\epsilon$ flow curves, which represent thermally activated processes that take place in the material being deformed and determine the change in structure, in particular with regard to the size of austenite grain. The axisymmetric hot compression investigations were carried out on cylindrical research specimens of 7 mm in diameter and 8.4 mm in length using a Gleeble 3800 simulator.

The research specimens were heated in the simulator by the resistance method, in an argon atmosphere. To reduce the coefficient of friction, the investigation specimens’ faces were coated with graphite and tantalum foil. Continuous compression experiments were carried out to $\varepsilon = 1.2$ on research specimens austenitized in a temperature range of 1100÷1250°C and deformed in a temperature range of 800÷1250°C at a strain rate of approximately 1.0s$^{-1}$, 3.6s$^{-1}$, 9.55s$^{-1}$ and 15.9s$^{-1}$. Immediately after the compression investigation, the research specimens were cooled in water to freeze the effects of hot plastic deformation. The metallographic microscopic examinations were carried out on longitudinal microsections of hot compressed and water- or air-cooled research specimens.

The structure observations were carried out with an Olympus GX71 microscope at magnifications of up to approximately 2000x and optionally in polarised light. In order to reveal the primary austenite grains, etching with saturated aqueous solution of picric acid at 70°C was used. In other cases, the microsections were etched with a reagent

| Element | C | Mn | Si | P | S | Ti | Nb | V | Al | N |
|---------|---|----|----|---|---|----|----|---|----|---|
| wt %    | 0.16 | 1.48 | 0.29 | 0.030 | 0.017 | 0.004 | 0.037 | 0.002 | 0.010 | 0.0098 |

Table 1. Chemical composition of HSLA steel under investigation
(nital). The measurement of the average diameter of the primary austenite grain was performed according to norm ASTM E112-10 [46] by counting the number of intersections on at least seven representative fields of observation. The results of these measurements were developed statistically for a confidence interval of $P=0.95$. Were carried out to determine the structure of austenite inherited by martensite by using a scanning electron microscope Inspect F with a Schottky field emission gun.

**RESULTS**

In the conducted experiments, the steel microalloyed with Ti, Nb and N, deformed in high-temperature compression investigation to $\varepsilon=1.2$, revealed flow curves of different waveforms, dependent mainly on the austenitizing temperature as well as the strain temperature and rate (Figure 1).

After austenitizing at $T_A=1250°C$ and $1200°C$ and deformation in a temperature range of $T_{\text{def}}=1100÷1250°C$ at a rate between $1.0s^{-1}$ and $15.9s^{-1}$, the recorded $\sigma$-$\varepsilon$ curves are characterized by the occurrence of more or less distinct maximum flow stress and a limited scope of fixed stresses, determined by the course of dynamic recrystallization (Figure 1–3).

At a lower deformation temperature ($900$ and $1000 °C$), dynamic recrystallization is not seen (Figure 1a, 1b). As the deformation ($\varepsilon = 1.2$) achieved in the compression experiment performed is too small to trigger the dynamic recrystallization process in the investigated steel. Under such deformation conditions, dynamic recovery is the decisive process that removes the effects of strain hardening.

At a higher deformation temperature ($1100÷1250 °C$), the $\sigma$-$\varepsilon$ curves show a slight reduction in flow stress value (Figure 3). The $\varepsilon_{\text{m}}$ values on the $\sigma$-$\varepsilon$ curves decrease as the strain temperature increases and the strain rate decreases. The changes in the flow stress of the investigated hot-deformed steel depend significantly on the strain temperature and strain rate, but do not depend clearly on the austenitizing temperature within the investigated range ($1250÷1100 °C$). The maximum stress values in the steel after austenitizing and deformation at $1200 °C$ increase from $78.8 \text{ MPa}$ to $115.6 \text{ MPa}$ along with increase in the strain rate in the range between $1.0 \text{ s}^{-1}$ and $15.9 \text{ s}^{-1}$ (Figure 2b). After austenitizing at $1200°C$
°C and deformation at 1100 °C while at the same maintaining the same rate, the $\sigma_{\text{max}}$ values increase from 108 MPa to 151 MPa (Figure 2a).

The metallographic examinations carried out allowed the effect of high-temperature deformation parameters on the size and shape of the primary austenite grain ($d_0$) before a $g \rightarrow a'$ transition conditioned by the course of thermally activated processes to be determined. It was found that austenitizing temperature had a decisive impact on the austenite grain size before high-temperature deformation (Figure 4). The investigated steel austenitized at 1100 °C reveals austenite grains of $d_0 = 36 \mu$m (Figure 4a). The increase in austenitizing temperature up to 1200 °C or 1250 °C results in an increase in the primary grain size $\gamma$ up to approximately 76 $\mu$m or 83 $\mu$m, respectively (Figure 4c). After the steel has been cooled down from this austenitizing temperature range in water, lath martensite with bainite areas (Figure 4b, d) and various lath sizes were revealed in the microstructure of the investigated steel.

The microphotograph (Figure 5) shows the results of metallographic observations of microalloy steel that has been deformed plastically in compression investigation at a strain rate of approx. 1.0 s$^{-1}$ and a constant strain degree ($\varepsilon = 1.2$) in a temperature range of 1200÷800 °C after austenitizing at 1200 °C. Primary austenite grains of varying sizes and forms depending on strain temperature were revealed in the structure of HSLA steels. Deformation at 1200 °C and 1100 °C allows the dynamically recrystallized $\gamma$ grains of approx. 16.1 $\mu$m (Figure 5a) and approx. 10 $\mu$m (Figure 5b), respectively, to be obtained within the entire material volume (Table 2).

The advantageous effect of refining $\gamma$ grains while maintaining their high dimensional uniformity and equiaxiality is shown clearly in the microphotograph (Figure 5b). After deformation at 1000 °C, fine recrystallized $\gamma$ grains were revealed in the near-boundary areas and occasionally within the primary austenite grains as a result of the initiation of dynamic recrystallization (Figure 5c), while after deformation at 800 °C, clearly elongated primary austenite grains with a number of deformed twins and slip bands, which are characteristic of the hardening stage on $\sigma$-$\varepsilon$ flow curves with a small contribution of thermally-activated processes, primarily dynamic recovery, were revealed (Figure 5d).

The additive effect of strain temperature and strain rate in the austenitizing temperature range (1250 °C, 1200 °C) on the structure of primary austenite grains of the investigated steel is shown.
The increase in strain rate up to approx. 15.9 s\(^{-1}\) causing the effect of delayed dynamic recrystallization of the investigated steel, recorded on the flow curves, also affects the microstructure of primary \(\gamma\) grains in the analyzed strain temperature range (Figure 6). At a comparable strain temperature of 1100 °C and strain degree (\(\varepsilon = 1.2\)), the primary austenite grains of the investigated steel deformed at a rate 1.0 s\(^{-1}\) are completely dynamically recrystallized (Figure 5b), while those deformed at a rate 3.6 s\(^{-1}\) only show partial dynamic recrystallization (Figure 7a). A similar effect of changes in microstructure of primary \(\gamma\) grains is observed for a strain temperature of 1000 °C and a strain rate of 3.6 s\(^{-1}\) or 15.9 s\(^{-1}\) (Figure 6). In (Figure 7b), austenite after dynamic deformation\((T_A = 1200°C, T_{\text{def}} = 900°C, \varepsilon = 1.2)\) reveals a microstructure of elongated grains with deformed twins inside them and fine dynamically recrystallized grains mostly at the boundaries of former primary

![Figure 4](image.png)

**Figure 4.** Structure of the investigated steel after austenitizing at: a, b) 1100 °C; c, d) 1250 °C; primary austenite grain size (a, c), martensitic – bainitic structure (b, d)

![Figure 5](image.png)

**Figure 5.** The effect of strain temperature at a strain rate = 1.0 s\(^{-1}\) on the structure of primary austenite grains of the investigated steel: a) 1200 °C, b) 1100 °C, c) 1000 °C, d) 800 °C \((T_A = 1200 °C, \varepsilon = 1.2)\)
austenite grains, which is a structure characteristic of the initial phase of dynamic recrystallization.

In the investigated steel deformed from ε = 0.268 at an austenitizing temperature of 1200 °C and a rate 1.0 s⁻¹, fine recrystallised austenite grains were revealed on the background of the primary γ grains corresponding to the applied austenitizing temperature (Figure 8a). Under such conditions, the strain of ε = 0.4 at 1000 °C is not sufficient to initiate dynamic recrystallization (Figure 8b). All the more in case of a compression investigation of the investigated steel at 800 °C and 900 °C and strain degree ε = 0.6, the revealed primary austenite grains only show distinct effects of strain hardening or dynamic recovery, but do not reveal the common symptoms indicating a potential contribution of dynamic recrystallization (Figure 9).

The austenite grain size for the analyzed range of strain parameters due to which the main process resulting in the reduction of hardening in HSLA steel was dynamic recrystallization is shown in Table 2. The structure of austenite inherited by martensite in the investigated steel after compression deformation at 1000 °C and a rate of 1.0 s⁻¹ (Tₐ = 1200 °C) is shown in the microphotograph (Figure 10). The boundaries of dynamically recrystallized austenite grains were observed in the background of martensite plates.

**CONCLUSIONS**

The austenitizing temperature before the γ to α' transformation has a decisive impact on the primary austenite grain size in HSLA steel. The investigated steel austenitized at 1100 °C is characterised by the austenite grains with average diameter of dₒ = 34 μm, and after austenitizing at 1200 and 1250 °C by grains of dₒ = 74 μm and 83 μm, respectively. For the investigated microalloyed steel with Ti, Nb and N, the size of the dynamically recrystallized γ grain decreases as the strain temperature is lowering and increasing. These parameters have a much greater impact on the regrouping and distribution of dislocations resulting in the formation of recrystallization.

**Table 2. Average primary austenite grain diameter**

| Austenitizing temperature, °C | Average grain diameter dₒ, μm | Strain temperature, °C | Strain rate, s⁻¹ | Average grain diameter d, μm |
|------------------------------|-------------------------------|------------------------|-----------------|----------------------------|
| 1200                         | 76.1 ± 12                     | 1200                   | 1.0             | 16.1 ± 2                   |
| 1100                         | 34.3 ± 10                     | 1100                   | 3.6             | 9.8 ± 2                    |

![Figure 6](image6.jpg)

**Figure 6.** Partially dynamically recrystallized primary austenite grains of the HSLA steel after deformation at 1000 °C and εₒ = 15.9 s⁻¹ (Tₐ = 1250 °C, ε = 1.2)

![Figure 7](image7.jpg)

**Figure 7.** Partially dynamically recrystallized primary austenite grains after deformation at εₒ = 3.6 s⁻¹ and a) 1100 °C, b) 900 °C (Tₐ = 1200 °C, ε = 1.2)
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Figure 8. The effect of HSLA steel strain degree and strain temperature on the structure of primary austenite grains: a) $\varepsilon = 0.268$, $T_{\text{def}} = 1200 \, ^\circ\text{C}$, b) $\varepsilon = 0.4$, $T_{\text{def}} = 1000 \, ^\circ\text{C}$ ($T_A = 1200 \, ^\circ\text{C}$, $\varepsilon^0 = 1.0 \, \text{s}^{-1}$).

Figure 9. The effect of the HSLA steel strain degree and strain temperature on the structure of primary austenite grains: a) $\varepsilon = 0.6$, $T_{\text{def}} = 800 \, ^\circ\text{C}$, b) $\varepsilon = 0.6$, $T_{\text{def}} = 900 \, ^\circ\text{C}$ ($T_A = 1200 \, ^\circ\text{C}$, strain rate = 1.0 s$^{-1}$).

Figure 10. Structure of dynamically recrystallized austenite compared to martensite after deformation of HSLA steel at 1000 °C and a rate of 1 s$^{-1}$ followed by water cooling ($T_A = 1200 \, ^\circ\text{C}$, $\varepsilon = 1.2$).

Figure 11. Result of the EBSD analysis carried out for the HSLA steel after deformation at 1000 °C and a rate of 1.0 s$^{-1}$ ($T_A = 1200 \, ^\circ\text{C}$, $\varepsilon = 1.2$ water).

fronts (grain refinement) than on the rate of their migration (grain growth). The reduction in a strain temperature from 1200 to 1100 °C at an austenitizing temperature of 1200 °C and strain rate of 1.0 s$^{-1}$ allowed the $\gamma$ grain size to be reduced from approx. 16 μm to approx. 10 μm. The increase in strain rate up to $= 3.6 \, \text{s}^{-1}$ at a strain temperature and an austenitizing temperature of 1100 °C has resulted in the reduction in austenite grain size to $d = 9.8 \, \mu\text{m}$ in HSLA steel. The grain size obtained during dynamic recrystallization does not depend on the primary grain size obtained during austenitizing in the analysed temperature range (1100–1250 °C).
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