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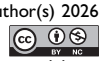
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Influence of hydrostatic pressure on the austenite grain growth and subsequent phase transformation of hot work steel AISI H13 during hot isostatic pressing

Yuanbin Deng[1,2], Jiali Zhang[1], Anke Kaletsch[1,2], Sebastian Weber[3] and Christoph Broeckmann[1,2]

Abstract

Newly developed hot isostatic pressing (HIP) units with integrated rapid cooling systems that are capable of operating at gas pressures up to 200 MPa provide the opportunity to densify and harden the metallic materials in a single step. For the application of these advanced HIP units in producing steels with defined microstructure, a crucial factor is the austenite grain size, which strongly influences the phase transformation during cooling and is effectively affected by the pressure during HIP. To study the effects of pressure, temperature and holding time on the growth of austenite grains and subsequently the final microstructure, a series of interrupted HIP experiments on hot work steel AISI H13 were conducted using such an advanced HIP unit. The austenite grain growth during HIP was analysed by metallographic microstructural investigations after rapid cooling. Thereafter, the effect of the austenite grain size on the phase transformation was studied using quenching dilatometry. Following this, the relationship between austenite grain size and martensitic/bainitic phase transformation temperature was determined, which indirectly indicates the influence of temperature, pressure and holding time on the final microstructure. By implementing this relationship into numerical models, the effect of HIP parameters on the microstructure of large-scale components, particularly those that experience large temperature gradients during the heating and cooling stages, can be predicted in the future.

Keywords

hydrostatic pressure, austenite grain growth, phase transformation, hot work steel, AISI H13, hot isostatic pressing

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Introduction

A newly developed hot isostatic pressing (HIP) unit with an integrated rapid cooling system, which can work at a gas pressure up to 200 MPa, allows the densification and hardening of the materials in a single process step. A major challenge for further application of the HIP with integrated rapid cooling in steel production is the effect of pressure on the component microstructure before rapid cooling. This requires in-depth understanding and clarification, as it will directly determine the final microstructure and consequently the properties of the steel, especially for the steels that undergo martensitic transformation, like martensitic tool steels. However, to the best of authors' knowledge, research on the microstructural evolution associated with the HIP pressure is still very limited.

Research activities on the influence of very high pressures in the range of one to several GPa on the iron-based microstructures already started in the 1950s, where the pressure was not applied via compressed gas but mechanical compression.[1] Nowadays, the isostatic pressure applied during HIP is usually discussed in the literature referred to the pure hydrostatic stress state in the material to emphasise the physical driving force for the microstructural evolution. Strong influences of the hydrostatic pressure on the transformation and precipitation behaviours of the Fe-C system during cooling were demonstrated. Several studies have shown that the application of the pressure shifts the transformation lines of both pearlite and bainite to lower temperatures and longer transformation times on isothermal and continuous cooling transformation (CCT) diagrams.[2–4] Hillliard and Cahn revealed that under a pressure of 3.4 GPa, the austenite-pearlite transformation experience an obvious decrease of transformation rate.[5] Angré et al. demonstrated that even a

[1]RWTH Aachen University, Institute for Materials Applications in Mechanical Engineering, Aachen, Germany
[2]Institute of Applied Powder Metallurgy and Ceramics (IAPK) at RWTH Aachen e.V., Aachen, Germany
[3]Ruhr Bochum University, Bochum, Germany

Corresponding author:
Yuanbin Deng, Institute for Materials Applications in Mechanical Engineering (IWM), RWTH Aachen, Augustinerbach 4, Aachen 52062, Germany.
Email: y.deng@iwm.rwth-aachen.de

Table 1. Chemical composition of the gas atomised AISI H13 powder.

	Mass fraction [wt.%]						
Elements	C	Si	Mn	Cr	Mo	V	Fe
This study	0.38	0.91	0.35	5.1	1.25	0.98	bal.
Specification	0.35–0.42	0.80–1.20	0.25–0.5	4.8–5.5	1.2–1.5	0.85–1.15	bal.

low HIP pressure like 170 MPa could already effectively suppress the intensity of the austenite-to-pearlite phase transformation and decrease the transformation velocity.[6] Evidence has also been given in the literature that not only the diffusion-controlled phase transformation, but also the diffusionless phase transformations like austenite to martensite transformation can be influenced by an applied pressure. Radcliffe and Schatz reported that under an isostatic pressure of 4.2 GPa, the martensite start temperature (M_S) was decreased by 150–200 °C depending on the alloy composition.[7] Consequently, steel classes would require more energy input to trigger martensitic hardening under the action of high pressures.

The recrystallisation and grain growth mechanisms of metallic alloys under the effect of pressure are another important topic besides the phase transformation. Some research has focused on the grain size evolution under hydrostatic pressure during HIP processes.[5,8–10] Tanner and Radcliffe found that a pressure of 4.2 GPa retarded recrystallisation during annealing of cold-rolled copper. For grain growth, they also found that the higher the applied hydrostatic pressure, the more significant the decrease in grain boundary mobility.[11] A similar conclusion has also been drawn by Krawczynska et al.,[9] who performed annealing treatments on nanograined austenitic stainless steel 316LVM under high hydrostatic pressures of 2 and 6 GPa and revealed that both recrystallisation and grain growth were strongly retarded by pressure. Furthermore, Qin et al. confirmed that the effect of hydrostatic pressure on recrystallisation during austenitisation is also not negligible.[12] They observed that a hydrostatic pressure of 150 MPa by HIP led to a finer microstructure in AISI M50 and claimed that hydrostatic pressure during annealing effectively slowed down the diffusion processes due to an increase of the activation energy for diffusion, which in turn retarded the recrystallisation.

So far, most studies have focused on the effects of mechanically applied pressure of 1 GPa on the phase transformation and grain growth. For martensitic tool steels that render wide application and are normally produced by casting and forging or powder HIP, the grain growth during HIP and its effect on the transformation of the microstructure during cooling are still unclear. In order to clarify the influence of hydrostatic pressure and temperature on the microstructural evolution during HIP, hot work steel AISI H13, as a member of the martensitic tool steels, was used as the powder material to study the austenite grain growth under different hydrostatic pressures, temperatures and holding durations, as well as the influence of austenite grain size on phase transformation during rapid cooling. Following this, the relationship between austenite grain size and martensitic/bainitic phase transformation temperature can be determined, which indirectly indicates the influence of temperature, pressure and holding time on the final microstructure.

Experimental procedures

Powder and capsule materials

Gas atomised powder of hot work steel AISI H13 (DIN X40CrMoV5-1, DIN No. 1.2344), produced by Carpenter Powder Productions AB, was used in this study. The powder exhibited a spherical morphology, with particle size distribution values of D_{10} of 16.54 μm, D_{50} of 83.51 μm and D_{90} of 236.06 μm. No inclusions or impurities were detected in the powder samples.

The chemical composition determined by optical emission spectroscopy measurements matches the specification for AISI H13, as shown in Table 1. The powder particle size is up to 500 μm. Stainless steel 304 (DIN X5CrNi18-10, DIN No. 1.4301) was used as the capsule material.[13]

Materials produced by HIP

In this study, the capsule geometry that is shown in Figure 1 was used. After the capsule manufacturing and the package tests, all capsules were filled with the powder in the same way: after being filled into the capsule, the powder was first pre-consolidated by a vibration procedure with a frequency of 60 Hz for 15 min, then evacuated with a pressure of 10^{-3} mbar for 24 h and subsequently sealed by forging and welding before undergoing HIP.

A series of HIP cycles with integrated rapid cooling were carried out in the HIP unit QIH-9 (Quintus Technologies, Sweden). As summarised in Table 2, in total 10 HIP cycles were performed, with different combinations of pressures

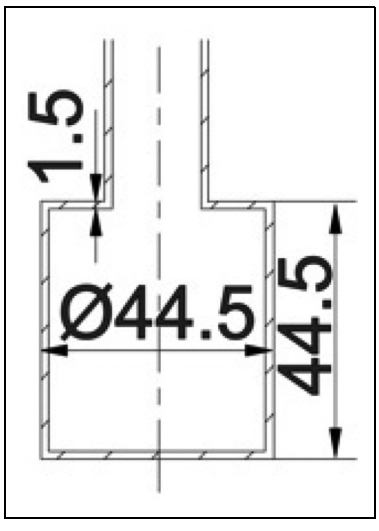

Figure 1. Technical drawings of the small lab capsule (unit: mm).

Table 2. List of combinations of the parameters applied in the HIP cycles.

No. of HIP cycle	Temperature [°C]	Pressure [MPa]	Holding time [h]
1	1050	100	0
2		100	2
3		170	
4		100	4
5		170	
6	1150	100	0
7		100	2
8		170	
9		100	4
10		170	

of 100 and 170 MPa, holding temperatures of 1050 and 1150 °C, and holding times of 0, 2 and 4 h. In all the interrupted cycles, the capsules were quenched after the heating or holding stage directly by gas in the HIP unit, which helped to ensure no occurrence of unexpected phase transformation. Due to the small size of the capsule, no large temperature gradient was expected; thus, the cooling rate in the centre of the capsule should be close to the region near the surface. Therefore, subsequent to the interrupted HIP cycles, each capsule was cut through the cross-section, and a cubic specimen of approx. $10 \times 10 \times 10$ mm^3 was machined from the centre of the half capsule by electrical discharge machining (EDM) to determine their densities based on Archimedes' principle and to study the microstructural evolution. In this way, the evolution of the relative density during the entire HIP cycle can be monitored and correlated to the microstructural evolution.

Microstructural characterisation

Prior austenite grains in hot work steel AISI H13. The focus of the study on producing hot work steel AISI H13 with targeted martensitic structure by HIP with integrated rapid cooling can only be achieved following an in-depth understanding of the influence of hydrostatic pressure on the prior austenite grain size (PAGS) and the martensitic transformation kinetics. To visually inspect the austenite grain growth under pressure during the HIP process, all the cubic specimens were hot mounted by conductive embedding materials (Polyfast, Struers ApS) after non-destructive density measurements. These embedded specimens were metallographically prepared following the general procedure of grinding with SiC papers, stepwise polishing using diamond suspensions, and final polishing with the suspension of alumina particles with a size of around 50 nm. After each polishing step, the specimens were cleaned in an ultrasonic bath and wiped with cotton to prevent cross-contamination between different steps.

The microstructures were studied in a FEI NanoLab G3CX Dualbeam (ThermoFisher) scanning electron microscope. The backscattered electron and secondary electron images were taken at an accelerating voltage of 15 kV. Electron backscatter diffraction (EBSD) measurements were performed using a Hikari XP II EBSD camera from Ametek-EDAX. The step size for high-resolution EBSD measurements was 50 nm. The EBSD results confirmed fully martensitic microstructure of all specimens. The prior austenite grains (PAGs) were reconstructed from the EBSD measurements by employing the ARPGE software.[14] This reconstruction process utilised the neighbour-to-neighbour method, which relies on the groupoid structure formed by the variants and their operators. Initially, within an experimental EBSD dataset, a triplet of neighbouring grains satisfying the groupoid composition table is sought with a stringent angle tolerance ($\leq 3°$). Subsequently, this identified "nucleus" was expanded by considering its neighbouring grains as potential candidates, retaining them only if they meet certain coherence criteria. The iterative "nucleation/growth" procedure continued until no further new nuclei could be discerned. This approach was characterised by its efficiency and robustness, facilitating the identification of a significant number of parent austenite grains. It should be noted that 20 areas with a size of around 100×100 μm^2 were measured by EBSD and employed for the reconstruction of the PAGs of each sample to ensure sufficient statistics. This is attributed to the long scan time of each area due to the small scan step size of 50 nm required for sufficient resolution. In this way, the PAGS was statistically obtained by analysing these reconstructed EBSD measurements.

Evolution of the phase transformation in the hot work steel. Dilatometry is one of the most precise methods to investigate phase transformations, as the allotropic transformations in the materials are usually associated with a change in specific volume. By fitting the measured volume changes with the corresponding phase transformation models, the phase transformations can be described mathematically. Following this, a quenching/deformation dilatometer of the type DIL805A/D from Bähr Thermoanalyse GmbH was employed to determine the phase-dependent thermal expansions and the volume changes resulting from the martensitic transformations in the hot work steel during cooling. To study the effects of the PAGS on the martensitic transformation, materials that were produced by the HIP cycles from No. 1 to 10 were selected, as these materials show a series of PAGS induced by different holding temperatures under different pressures with different holding times.

The specimens from HIP cycles 1, 6, 8, 9, and 10 were first manufactured to the required geometries (diameter: 4 mm, height: 10 mm) by EDM and subsequently mounted in the dilatometer to be inductively heated up to an austenitising temperature of 1050 °C. The temperature was monitored by a thermocouple (type S), which was welded on the middle section of each specimen surface. The specimens were continuously heated to the austenitising temperature within 5 min, held for 10 min, and then quenched by different cooling rates between −30 and −0.05 °C/s to room temperature. The phase transformations from austenite to martensite and/or bainite during the quenching were recorded and investigated by analysing the length change of each specimen.

Results and discussion

Influence of temperature, pressure and holding time on the austenite grain growth

The microstructure of the consolidated specimens of hot work steel AISI H13, after various HIP cycles, was

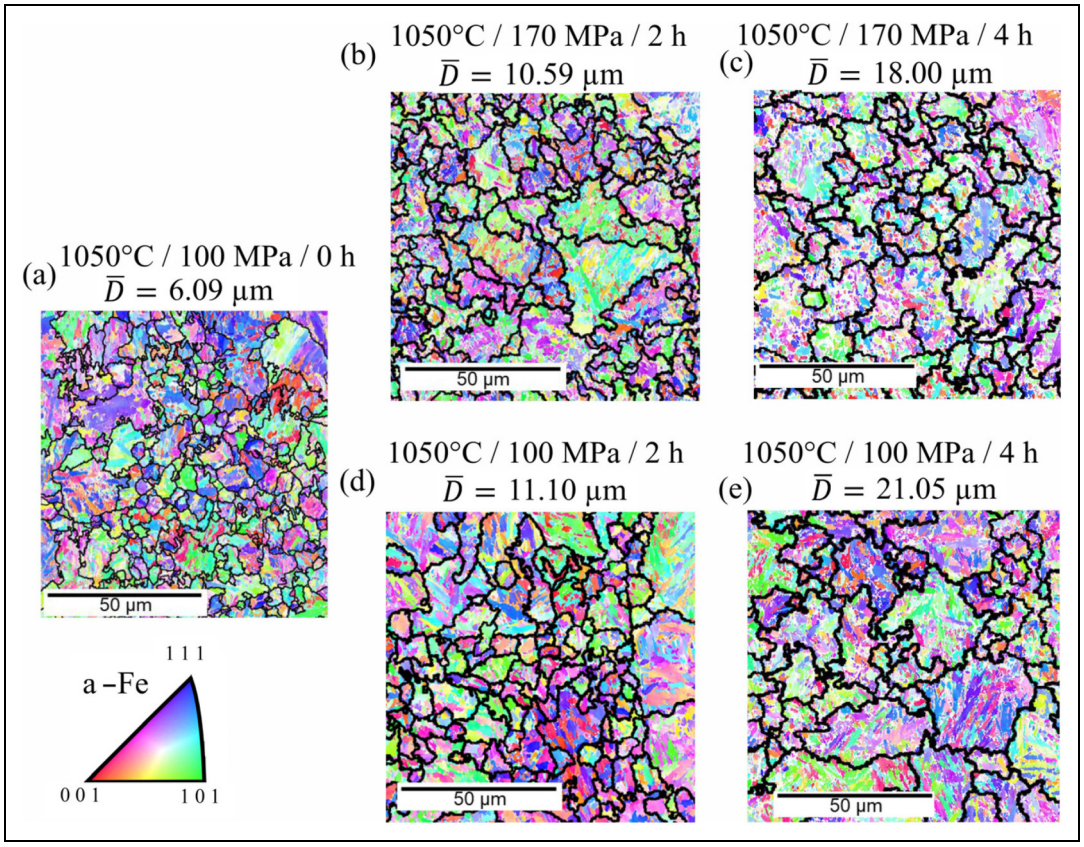

Figure 2. EBSD inverse pole figure (IPF) maps with black colour labelled reconstructed austenite grain boundaries under various pressures at 1050 °C: initial state without holding time (a), HIPed states under 170 MPa for 2 h (b), 4 h (c) and under 100 MPa for 2 h (d), 4 h (e).

characterised using the methods described in section 'Microstructural characterization'. Figures 2 and 3 present the EBSD inverse pole figure (IPF) maps of all investigated specimens with the black coloured lines labelling the PAG boundaries reconstructed using ARPGE.[14] By measuring more than 300 PAGs on each specimen, the PAGS distribution was determined, as shown in Figure 4.

Figure 2 shows the microstructure of all the specimens which were HIPed at 1050 °C. As seen in Figure 2(a), when the HIP temperature reached 1050 °C, the powder was already densified in the capsule without any holding time, as no obvious pores were observed in the EBSD IPF map. As the holding time increased, the PAG grew at 1050 °C under a pressure of 100 MPa. After a HIP process of 2 and 4 h, the average PAGSs reached 11.10 and 21.05 µm, as shown in Figure 2(d) and (e). Increasing the HIP pressure to 170 MPa only slightly limited the grain growth, resulting in smaller grain sizes after 2 and 4 h of holding time (Figure 2(b) and (c)) compared to those obtained at 100 MPa (Figure 2(d) and (e)).

Figure 3 shows the evolution of the PAGS at the holding temperature of 1150 °C under different pressures and with different durations of the holding stage. Compared to the IPF map shown in Figure 2(a), it can be seen that by keeping the pressure at 100 MPa, the average PAGS increased to 12.68 µm when the holding temperature was elevated to 1150 °C. This PAG size was established as the initial condition for investigating the growth of PAG at different pressures with various holding times at 1150 °C. In Figure 3(b) to (c), it is evident that at 1150 °C, austenite grains underwent significant growth with increasing holding time. Comparing the two different hydrostatic pressures applied at 1150 °C, the growth of the austenite grains was increasingly restricted as the pressure increased from 100 to 170 MPa. After holding at 1150 °C for 4 h, the average PAGS reached 45.2 µm at 100 MPa. However, increasing the pressure to 170 MPa resulted in a smaller average grain size of 30.5 µm.

Figure 4(a) shows the PAGS distributions of the specimens HIPed with different combinations of temperatures (1050 or 1150 °C) and pressures (100 or 170 MPa) for a holding time of 2 h. The specimens HIPed at 1050 °C under 100 and 170 MPa exhibit relatively narrow PAGS distributions with closely aligned peaks. However, when the HIP temperature is increased to 1150 °C while keeping the pressure unchanged, the PAGS distribution becomes significantly wider, for instance, the blue versus red curve. When the holding time is extended to 4 h, a similar influence of the temperature can also be observed, as illustrated in Figure 4(b). As for the effect of pressure, the comparison between the green and red curves in Figure 4(a) already reveals that increasing pressure results in refinement of the PAGs, although this effect is not obvious for the specimens HIPed at 1050 °C for 2 h. However, this influence of the HIP pressure on suppressing the PAG growth becomes more pronounced when the holding time is extended to 4 h (Figure 4(b)). This demonstrates that by increasing both the HIP temperature and the holding time, the effect of pressure on grain growth becomes more apparent.

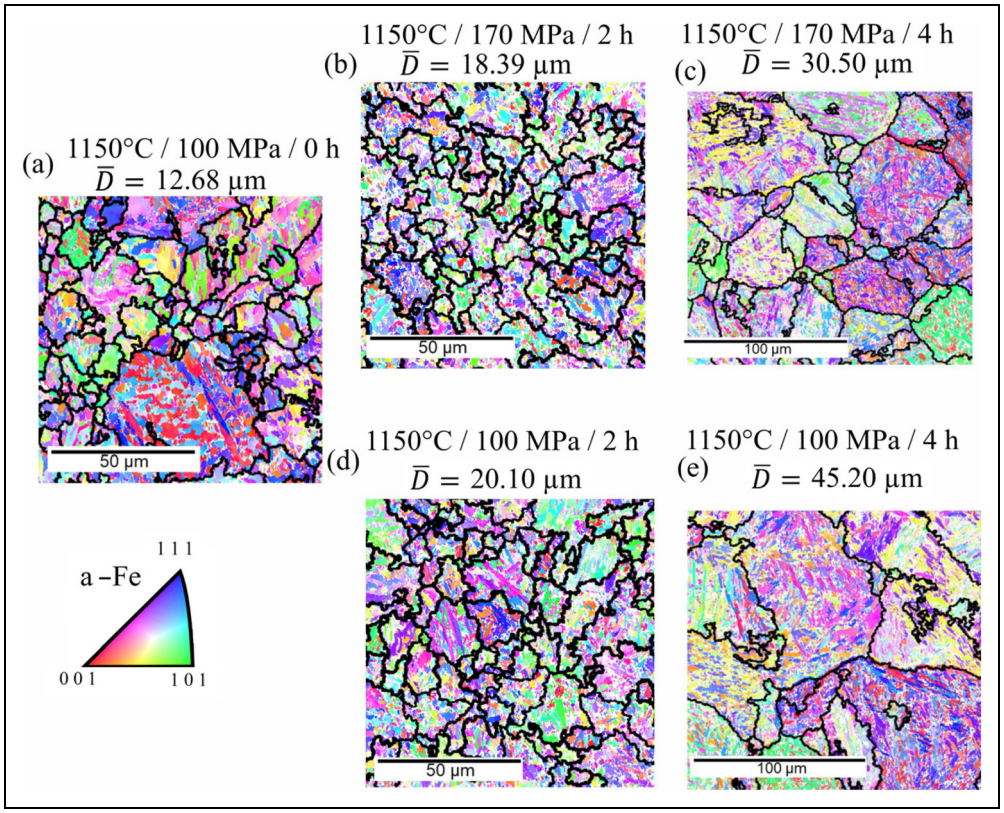

Figure 3. EBSD IPF maps with reconstructed austenite grain boundaries under two different pressures at 1150 °C: initial state under 100 MPa without holding time (a), HIPed states under 170 MPa for 2 h (b), 4 h (e) and under 100 MPa for 2 h (d), 4 h (e).

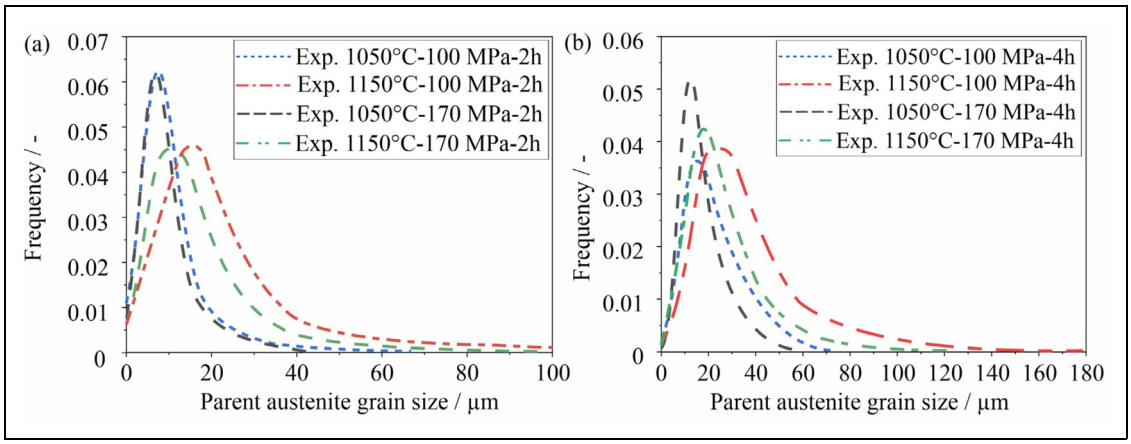

Figure 4. PAGS distributions of the specimens HIPed with different combinations of pressures (100 or 170 MPa) and temperatures (1050 or 1150 °C) for 2 h (a) and 4 h (b).

Influence of the austenite grain growth on the martensitic and bainitic transformation

Figure 5 shows the dilatometry curves determined during the cooling of the specimens in correlation with the PAGS, which clearly reveals that the M_S decreases as the PAGS decreases. The highest M_S of 346 °C was determined using the cooling curve of the specimen with an averaged PAGS of 45.20 µm, while the lowest M_S of 300 °C was determined on the specimen with an averaged PAGS of 6.09 µm. This inverse correlation between the PAGS and the M_S has been confirmed by many studies in the literature.[15,16] However, no key factor for this has been identified yet, as the refinement of PAGs causes a complex interplay among many factors during martensitic transformation. These factors include density of nucleation sites at the grain boundary, internal elastic strain due to different martensite lath morphologies, strain energy relaxation by self-accommodation of multivariant formation and so on.[16] Nevertheless, it is no doubt that the increase of HIP pressure cause refinement of PAGs which subsequently induce postponed onset of martensitic transformation (Figure 6).

To gain an in-depth understanding of the effect of PAGS on the phase transformation during cooling, the CCT diagram was obtained by analysing the dilatometry curves of the specimens with average PAGSs of 6.09 and 45.20 µm. The shift of the curves reveals that the PAGS influences not only the martensitic transformation, but also the bainitic transformation. As the PAGs coarsen, the onset of the bainitic transformation

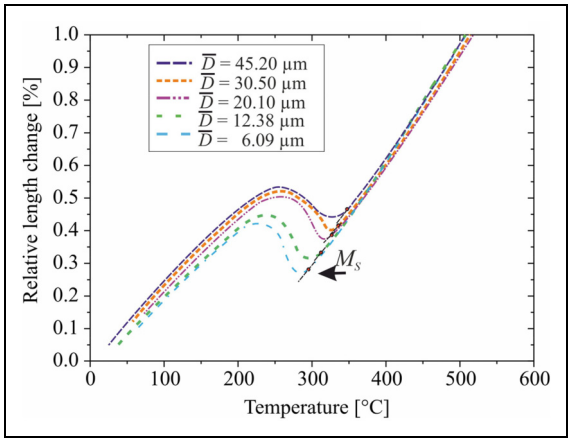

Figure 5. Dilatometry curves during cooling in correlation with the average PAGS.

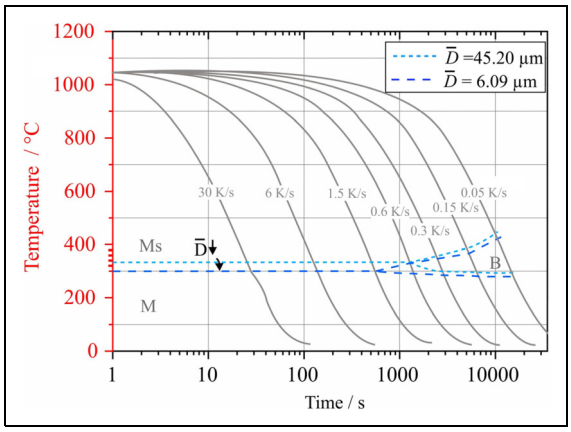

Figure 6. CCT diagram of the HIPed AISI H13 with two different average PAGS.

shifts to a later time point. In addition, the larger the austenite grains, the higher the bainitic transformation temperature is. Recently, Kang et al. and Lee et al.[15,17] investigated the effect of PAGS on phase transformations and their reported bainite volume fraction change by considering the decrease in bainite start temperature with decreasing PAGS showed good agreement with the CCT diagram determined in this study.

Conclusions

In this work, the influences of temperature, pressure and cooling rate on the austenite grain growth and the subsequent bainite/martensite transformation during HIP in the newly developed HIP unit with integrated rapid cooling were systematically studied. The main conclusions can be drawn as follows:

- The HIP unit with integrated rapid cooling allowed for the 'freezing' of the microstructure after the HIP process, which can be used to reconstruct the PAGs of the steels for a deeper understanding of the process parameters, for example, hydrostatic pressure, temperature and holding time, on microstructural evolution during the HIP process. Higher temperatures and longer holding times accelerate austenite grain growth, while higher isostatic pressure can partially suppress it.
- The martensite/bainite start temperatures in relation to the PAGS were determined by studying the thermal expansion in the dilatometer. Martensite/bainite start temperatures increase with the coarsening of the PAG. This provides insight into how the HIP process parameters indirectly influence the phase transformation during the cooling phase and the final microstructure.
- For a further in-depth study, these findings can be implemented into a numerical model to enable the prediction of the microstructural evolution in large components, especially those that experience large temperature gradients during the heating and cooling stages.

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ORCID iD

Yuanbin Deng https://orcid.org/0000-0002-5057-4876

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Declaration of conflicting interests

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