



Influence of metastable retained austenite on macro and micromechanical properties of steel processed by the Q&P process



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ABSTRACT

By stabilising metastable austenite with a suitable morphology in a martensitic structure, it is possible to impart to multi-phase steels high ductility combined with tensile strengths exceeding 2000 MPa. One way to achieve such mixed structures consisting of martensite and retained austenite (RA) is the Q&P (quenching and partitioning) process. The resulting structure contains metastable austenite in the form of thin foils located between martensite laths or plates. The stability of austenite under mechanical loading is the essential factor contributing to the extraordinary plasticity of such materials during cold deformation. A steel with 0.43% of carbon, alloyed with manganese, silicon and chromium was chosen for the experiment described in the present paper. Using the Q&P process, a martensitic structure with 20% of retained austenite was obtained. As cold plastic deformation causes the austenite to transform, 10% cold deformation was applied after the Q&P process. This deformation reduced the RA fraction to 11%. Materials prepared by this method were examined using micro-pillar compression experiments. Using the focused ion beam (FIB) method, pillars of $3 \times 3 \mu\text{m}$ cross-section and $8 \mu\text{m}$ length were fabricated. These were afterwards mechanically tested in situ in an electron microscope in quasi-static compression at a true strain rate of $3 \times 10^{-4} \text{ s}^{-1}$ to different amounts of plastic strain. The experiment showed that mechanical properties of the two conditions of material differ in terms of yield strength and the strain hardening exponent. An additional metallographic analysis of structures, including the exploration of the influence of decomposition of retained austenite, was performed.

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1. Introduction

In medium and high-carbon steels, very high strengths are typically achieved by inducing martensitic transformation, which is at the expense of the material's ductility. The ductility can, however, be improved subsequently, for instance, by tempering the martensitic microstructure. This, on the other hand, leads to a decline in strength. An alternative approach to controlling the properties of the material is to cause an additional microstructural phase to form. One such phase may be the retained austenite which transforms to strain-induced martensite during plastic deformation [1,2]. Its stability is an important factor, as it has a favourable impact on the strain hardening coefficient and on the strength and ductility of the material. Another factor is the austenite's ability to absorb dislocations from adjacent martensite needles, thus improving the deformation capacity of martensite during uniform deformation.

One of the techniques for preparing a microstructure of this type is a heat treatment method known as the Q&P process (Quenching and Partitioning). It is characterized by quenching the material from the austenitizing temperature to a region between M_s and M_f temperatures, where it is held to allow carbon to migrate from the oversaturated martensite to metastable austenite. As a result, the stability of austenite increases [3]. With suitable chemistry and processing parameters, ultimate strengths exceeding 2000 MPa and elongations above 10% can be achieved [4,5].

Mechanical properties of the martensitic–austenitic microstructure depend in part on the stability of its retained austenite (RA) component. Using the Q&P process, such microstructures typically contain between 10% and 15% RA. In most cases, retained austenite takes the form of thin films on the martensite lath boundaries. This sets steels treated by the Q&P process apart from TRIP steels, in which retained austenite is present in a granular form. The film morphology of retained austenite has a stronger influence on the elongation behaviour than the granular type [6]. The stability of RA depends on a number of aspects. In terms of chemical

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composition, it is the content of carbon and other alloying elements, such as manganese and silicon, which depress the M_s temperature below room temperature. The concentration of carbon in retained austenite should be higher than 1 wt.%. Where the carbon level is below 0.5 wt.%, austenite transforms to martensite very rapidly during plastic straining. By contrast, at concentrations above 1.8 wt.% austenite is very stable and survives the cold deformation [7]. Silicon is used to retard carbide precipitation and promote the diffusion of carbon to austenite. In addition to austenite stabilization, manganese improves carbon solubility in austenite and retards pearlite formation. Another suitable alloying element for Q&P steels is chromium [8]. It strengthens the solid solution, retards pearlite and bainite formation and improves the material's hardenability and resistance to tempering.

Austenite stability is also controlled by the size of its particles. The optimum size of austenite particles is in the range of 0.01–1 μm . The stability and strength of RA also depend on the surrounding phases [8,9]. Four transformation temperatures are important for the stability of retained austenite: M_s , M_s^σ and M_{d30} and M_d [8].

The M_s temperature can be determined using both experimental and empirical methods. In calculating the M_s value, a number of phenomenological models can be employed which account for the effects of alloying elements. These include, for instance, the Andrews' model [10] (Eq. (1)) and the model by Mahieu et al. [8] (Eq. (2)). As part of the development of the Q&P process, another empirical formula was constructed, taking into account the effect of the austenite grain volume V_γ [11] (Eq. (3)).

M_s^σ is the temperature below which retained austenite transforms to martensite under critical external stress. Above this temperature the transformation is induced by strain. At this temperature, the stress required to induce the transformation is equal to the yield stress of austenite. Above this temperature, the austenite undergoes deformation and the martensitic transformation is facilitated by plastic strain. In the region just above M_s^σ , the level of stress required for inducing the transformation remains constant. If the temperature rises above M_d , austenite remains stable and does not transform to martensite, regardless of plastic deformation. The M_{d30} temperature is the temperature at which 30% tensile strain causes 50% of austenite to transform to martensite. This parameter is widely used to describe the stability of austenite. It can be calculated using empirical formulas based on chemical composition [9] (Eq. (4)).

$$M_s(^{\circ}\text{C}) = 539 - 423\text{C} - 30.4\text{Mn} - 17.7\text{Ni} - 12.1\text{Cr} - 11\text{Si} - 7\text{Mo} \quad (1)$$

$$M_s(\text{K}) = 273 + 545.8 \cdot e^{-1.362\text{C}} - 30.4\text{Mn} - 7.5\text{Si} + 30\text{Al} - 59.9\text{P} \quad (2)$$

$$M_s(^{\circ}\text{C}) = 545 - 423\text{C} - 30.4\text{Mn} - 60.5V_\gamma^{-1/3} \quad (3)$$

$$M_{d30}(^{\circ}\text{C}) = 413 - 462(\text{C} + \text{N}) - 9.2\text{Si} - 8.1\text{Mn} - 13.7\text{Cr} - 9.5\text{Ni} - 18.5\text{Mo} \quad (4)$$

All research efforts to date have focused on the behaviour of such microstructures on a macroscopic scale and on describing their properties. This is why the present investigation is aimed at obtaining new findings and at describing the phenomena which arise from the deformation behaviour of these materials within a microscopic volume. One of the available techniques is micro-compression testing of micro-pillars with dimensions in the order of micrometers. Thanks to the pillar size, plastic straining and fracture propagation within a few martensite needles can be monitored in the test. According to literature sources, the presence of austenite delineating martensite needles can be expected. This is why the boundaries of needles should be observed at high resolution, as they are the locations where localized deformation and failure are expected to occur. Major attention was paid to comparing the deformation behaviour of microstructures formed by Q&P processing with various amounts of strain.

2. Experimental programme

The experimental programme consisted of macroscopic-scale deformation tests and investigation of the behaviour of steels containing martensite with retained austenite using microscopic-volume specimens. The experimental materials were Q&P processed steels. Micro-pillars made from these materials were subjected to compressive deformation. The compressive loading was monitored and the pillar deformation recorded by SEM imaging.

This experiment was performed on 42SiCr steel with 0.42% carbon, alloyed with silicon, manganese and chromium (Table 1). This chemistry was selected with regard to the ability to provide sufficient stability of retained austenite, solid solution strengthening and to retard cementite precipitation and bainite and pearlite formation. The initial microstructure with a hardness of 290 HV10 consisted of pearlite and a very small proportion of ferrite. Miniature tension tests revealed the material's strength of 981 MPa and elongation of $A_{5\text{mm}} = 30\%$.

2.1. Thermomechanical treatment

Two groups of specimens were prepared for testing using two treatment procedures in order to compare their properties: the Q&P process and Q&P + cold working. The Q&P process was carried out in a thermomechanical simulator. It consisted of austenitizing at 900 $^{\circ}\text{C}$ for 100 s and of 20-step incremental deformation with the accumulated true strain of $\phi = 5$, applied within a temperature interval of 900–820 $^{\circ}\text{C}$. The main purpose of the applied deformation was to refine the microstructure. The deformation was followed by cooling to 200 $^{\circ}\text{C}$, subsequent reheating to the partitioning temperature of 250 $^{\circ}\text{C}$ and holding for 600 s in order to stabilize metastable austenite by absorbing carbon which migrated from martensite. The quenching and partitioning temperatures were chosen with regard to the known M_s temperature (Table 2), which had been determined using dilatometer measurement at a cooling rate of 20 $^{\circ}\text{C}/\text{s}$. In addition, a verification simulation was performed using the JMatPro program (Version 6.2) and an additional verification calculation was carried out with the aid of empirical models (Eqs. (1)–(3)) (Table 2). The M_{d30} temperature was verified for the chemical composition in question using Eq. (4).

The second treatment procedure comprised the same Q&P process and an additional cold working step with 10% tensile strain. The cold working reduced the amount of retained austenite because part of the RA transformed to strain-induced martensite.

Table 1
Chemical composition of the experimental steel 42SiCr.

C	Si	Mn	Cr	Mo	Nb	P	S	Ni	N	UTS R_m (MPa)	Elong $A_{5\text{mm}}$ (%)	HV10 (–)
0.43	2	0.59	1.33	0.03	0.03	0.009	0.004	0.07	0.01	981	30	290

Table 2
 M_s and M_{d30} temperatures of the 42SiCr steel, as determined by various methods.

JMatPro		Dilatometry –20 $^{\circ}\text{C}/\text{s}$		Andrews	Mahieu et al.	Lee et al.	M_{d30} ($^{\circ}\text{C}$)
M_s ($^{\circ}\text{C}$)	M_f ($^{\circ}\text{C}$)	M_s ($^{\circ}\text{C}$)		M_s ($^{\circ}\text{C}$)	M_s ($^{\circ}\text{C}$)	M_s ($^{\circ}\text{C}$)	
298	178	289		299	272	322/205	167

2.2. Mechanical testing

The treated specimens were used for measuring mechanical properties associated with their resulting microstructures. Besides hardness measurements, miniature tensile tests were carried out to determine the material's bulk properties. Micro-compression tests on micro-pillars were conducted to measure and compare the local properties.

The tensile test was performed using miniature specimens with the gauge length of 5 mm and a cross-section of 2 × 1.2 mm. The strain rate applied was 10⁻² s⁻¹.

The in situ micro-compression test was performed in a Leo 982 scanning electron microscope (SEM) using the Unat-SEM indenter (Asmec) [12]. Preparation of samples for micro-pillars comprised steps shown in Fig. 1. The central part of a miniature tensile specimen was cut out and conventionally ground and polished to a thickness of ~120 μm. Subsequently, the grey shaded area in Fig. 1 was selectively electrochemically etched down to a thickness of a few μm. From this thin wedge, pillars with a cross-section of 3 × 3 μm and a length of 8 μm (Fig. 1) were made

using a focused ion beam (FIB) workstation. In the electron microscope chamber, the pillars were subjected to quasi-static compressive deformation introducing a strain rate of 3 × 10⁻⁴ s⁻¹ (Fig. 2). The loading process was captured as a video sequence, from which stills could be obtained for characterizing the pillar deformation process (Fig. 3). Technical stress–strain data was calculated using the initial cross section without correcting for the machine compliance. The value of the strain hardening coefficient was calculated using the Ramberg–Osgood relationship. Multiple tests with various amounts of plastic strain between 7.5% and 20% were conducted.

3. Results and discussion

The Q&P process led to a martensitic microstructure with a small amount of bainite and no free ferrite (Fig. 4). Using X-ray diffraction phase analysis, it was found that the material contained

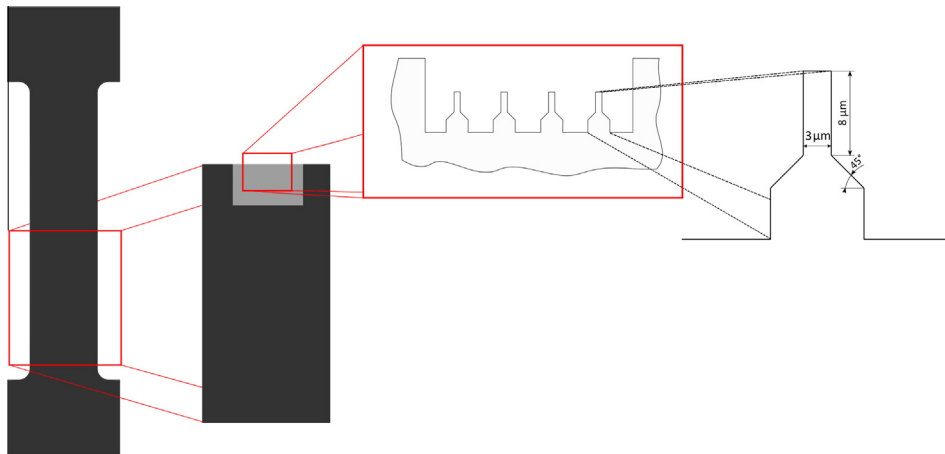


Fig. 1. Sampling and specimen preparation for the micro-compression test starting from a miniature tensile specimen.

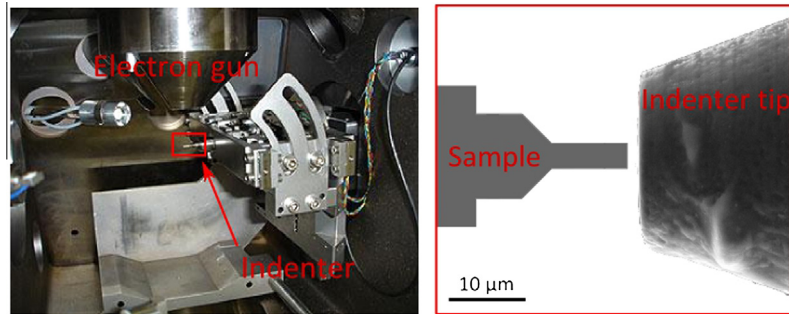


Fig. 2. Arrangement in the microscope chamber and the principle of the micro-compression test.

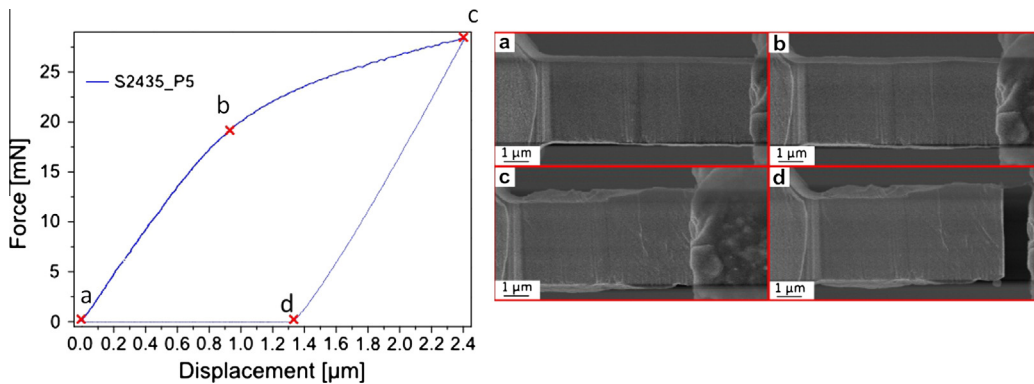


Fig. 3. Diagram of micro-compression test on Q&P – processed material and micrographs of pillar deformation upon introducing a displacement of 2.4 μm corresponding to a total plastic strain of 7.5%.

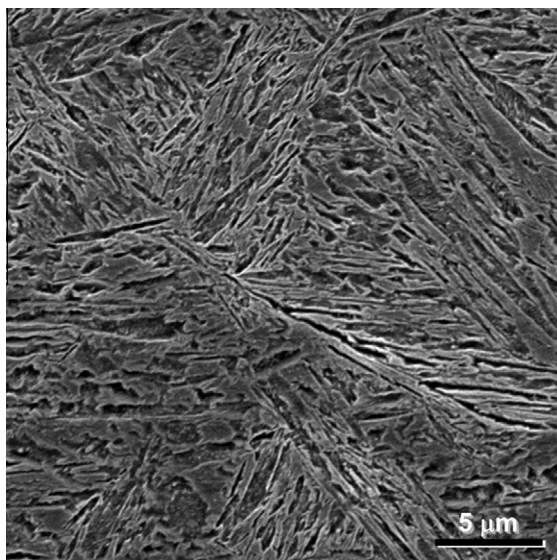


Fig. 4. SEM micrograph showing the microstructure of Q&P – processed material. Martensite with a small fraction of bainite and 20% RA, etched with Nital.

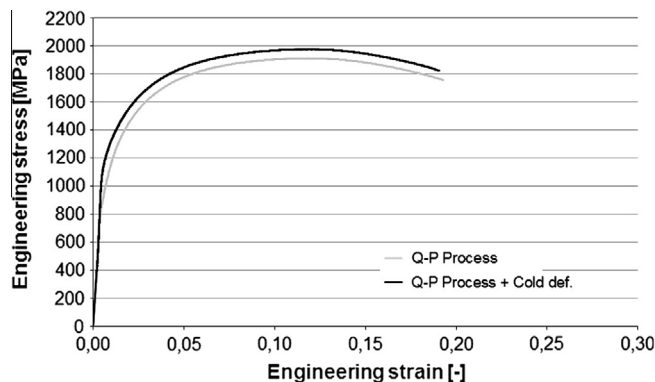


Fig. 5. Stress–strain curves of 42SiCr steel after both treatment methods.

20% retained austenite (Table 3). As no polyhedral austenite particles were found in the microstructure, it can be assumed that austenite is present in the form of thin films on the boundaries of the martensite needles. However, the presence of austenite in other additional locations cannot be ruled out.

The 10% strain introduced by cold working following the Q&P process reduced the retained austenite fraction by almost 9% to the final 11%. No substantial changes were found by metallographic examination in the nature of the microstructure. The cold working slightly raised the material's hardness from 585 HV10 to 600 HV10 (Table 3).

Results of the miniature tensile test show that the Q&P process led to a yield strength of 969 MPa, an ultimate tensile strength of 1907 MPa and an elongation of 17%. Cold working following the Q&P process increased the yield strength to 1210 MPa, as well as the ultimate strength to 1980 MPa, whereas the elongation values declined by a mere 2% (Fig. 5). Despite the prevalent martensitic microstructure, an unusually high elongation level was achieved, which was due to the volume fraction of plastic austenite. In both cases, the fracture surfaces with dimples indicated ductile fracture (Fig. 6). After the tension test, the retained austenite fraction was below the detectable limit for X-ray diffraction analysis, which means less than 3%. This suggests that the retained austenite decomposed into martensite in the course of cold deformation.

The micro-compression test was performed using micro-pillars with a size of $3 \times 8 \times 8 \mu\text{m}^3$. Several pillars (Fig. 1) were made from the specimens with Q&P processing and from Q&P + cold-worked specimens. The martensitic microstructure and a relatively high volume fraction of retained austenite led to surface quality problems: a wavy appearance of the surface of the FIB cut surfaces. Various milling current settings were tried within the range of 500–100 pA. Reducing the current to 100 pA improved the surface condition but the adjusted process required a much longer time to

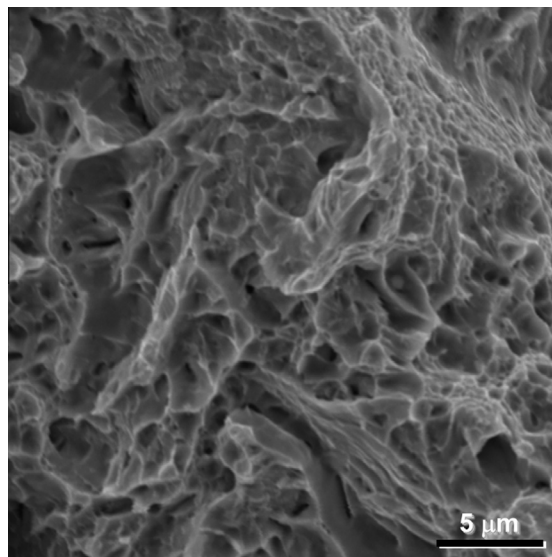


Fig. 6. SEM image of the fracture surface of a failed tensile specimen in Q&P + cold working condition, showing a dimple rupture mode of ductile fracture.

complete. Another difficulty associated with the pillar deformation was caused by the heterogeneous microstructure. Since only martensite laths were sampled in micro-pillars, the slopes of stress–strain curves recorded during loading and unloading varied (Fig. 7).

Micro-pillar tests of the material after a plain Q&P process were performed to different amounts of plastic strain (Table 4 and Fig. 7). Slip lines on the surface of the pillar began to emerge at a plastic strain as low as 7.5%. However, no pillars exhibited localized deformation or failed, even at the largest strain. The average yield strength was approx. 1448 MPa, and the strain hardening coefficient reached 4.56. No slip was detected at boundaries of martensite needles, although it was anticipated with regard to the presence of retained austenite. This suggests that the interaction between martensite and retained austenite may be more complex and that austenite may also be present within martensite needles.

Table 3
Mechanical properties upon thermomechanical treatment.

	RA (-)	HV10 (-)	Tension test			Compression test	
			0.2 PS (MPa)	UTS (MPa)	$A_{5\text{mm}}$ (%)	0.2 PS (MPa)	n (-)
Q–P	20	585	969	1907	17	1448	4.56
Q–P + cold worked	11	600	1210	1980	15	1705	3.37

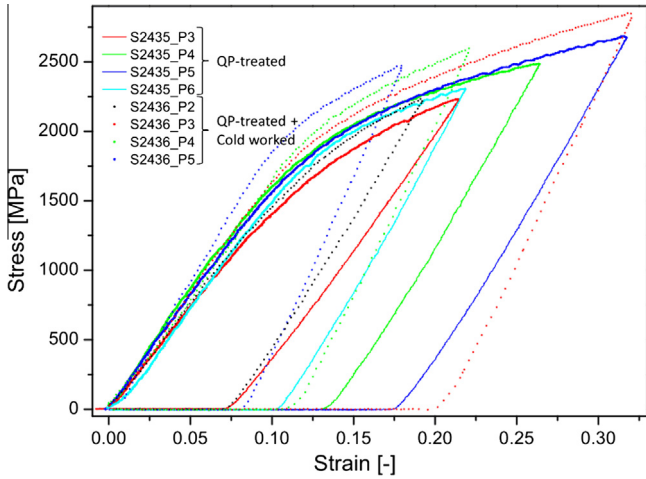


Fig. 7. Comparison between test plots for Q&P (S2435) material and the Q&P + cold worked (S2436) material for several pillars with various plastic strain magnitudes.

With the Q&P + cold worked material, plastic strain magnitudes of 7.5, 12 and 20% were applied (Table 4 and Fig. 7). Work hardening which was induced prior to the compression test was manifested by the increase in the yield strength to 1705 MPa and by the lower value of the strain hardening coefficient of 3.37. This effect of reduced plasticity can be seen in the test plots (Fig. 7). It was only at the 20% plastic strain when partial slip was observed and the pillar’s microdeformation became slightly non-uniform (Fig. 8).

Table 4
Micro-compression tests to various plastic strain levels.

Treatment	Plastic strain (%)	0.2 PS (MPa)	n (-)	Treatment	Plastic strain (%)	0.2 PS (MPa)	n (-)
Q-P	7.5	1350	4.17	Q-P + cold worked	7.5	1410	2.58
	13	1500	5.8		20	1650	3.48
	17.5	1542	4.83		12	1910	3.26
	10	1398	4.16		7.5	1850	4.15
	Mean value	1448 ± 77	4.56 ± 0.4		Mean value	1705 ± 196	3.37 ± 0.56

4. Conclusion

The Q&P processing of the low-alloyed steel produced martensitic microstructure with 20% retained austenite. Its yield strength, ultimate strength and elongation to failure were 969 MPa, 1907 MPa and 17%, respectively. The application of the Q&P process followed by cold working led to a microstructure with only half the volume fraction of retained austenite. Its yield strength was 1210 MPa and its ultimate strength reached 1980 MPa.

The amount of retained austenite was measured by X-ray diffraction. Despite its relatively high total volume fraction, the morphology and distribution of the retained austenite in the microstructure are impossible to identify using optical or scanning electron microscopy. To date, it has been presumed that retained austenite forms foil-like particles between martensite laths or plates, thus improving the plasticity of the otherwise brittle martensitic microstructure. In order to identify the mechanism of local deformation of this structure and to map its tendency to localized straining within a micro-volume, in situ micro-compression tests were performed on micro-pillars with a size of $3 \times 3 \times 8 \mu\text{m}^3$. These results showed that both microstructures in question are easily capable of plastic deformation. The pillars did not fail even at 20% plastic strain. The pillar surfaces exhibited signs of plastic deformation within martensite, as well as strain localization due to the inhomogeneous microstructure. The miniature tensile tests revealed the mean difference between the proof stresses of materials upon the two treatments is 257 MPa. This was not substantially different from the same parameter calculated from the pillar tests: 241 MPa. The pillars exhibited no slip localization in

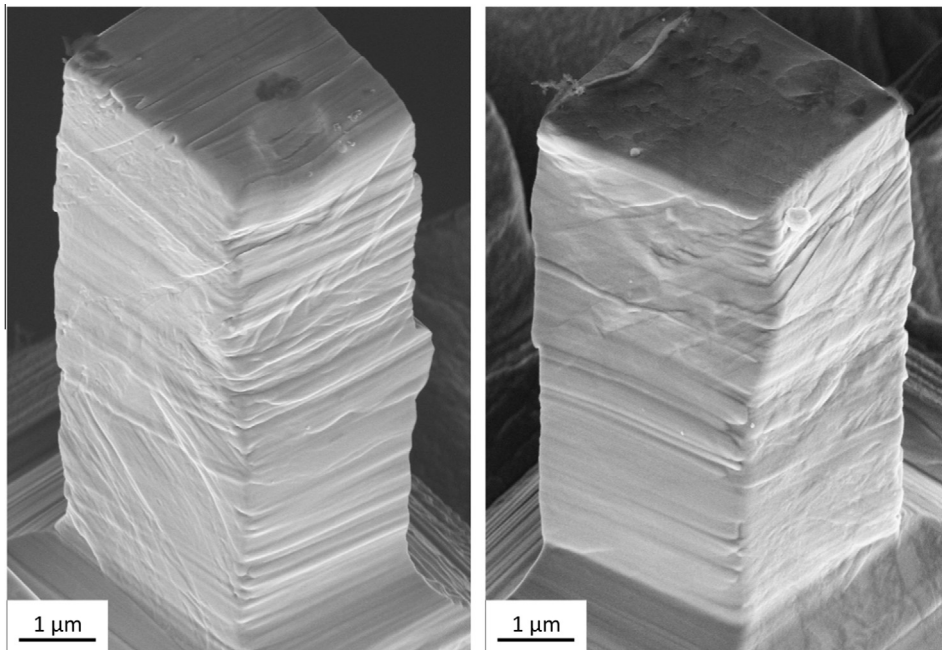


Fig. 8. Inclined SEM images of deformed pillars. The conditions were Q&P, 12% plastic strain (left), and Q&P + cold worked, 17.5% plastic strain (right).

austenite foils. The plastic strain typically occurred in multiple locations of the pillar. The results suggest that new hypotheses on the morphology of the microstructure in Q&P-processed martensitic–austenitic steels will have to be sought in the future.

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