



Martensite Fractions Measured by XRD on Fracture Surfaces of Austenitic Stainless Steels Tensile Tested in Gaseous Helium and Hydrogen

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Abstract

Although martensitic transformation is neither necessary nor sufficient to explain hydrogen effects in austenitic stainless steels, the formation of a second phase upon straining results in local strain incompatibilities within the microstructure which can result in hydrogen assisted crack initiation. The fraction of α' -martensite on fracture surfaces of ten commercial austenitic stainless steels of different austenite stability was measured by quantitative x-ray diffraction (XRD). The main results are: (1) for a given steel, martensite contents on fracture surfaces of specimens tested in helium were higher compared to those tested in hydrogen and (2) tensile reduction in area of the specimens tested in hydrogen decreases with increasing martensite content on the fracture surface.

Keywords Hydrogen embrittlement · XRD · Austenitic stainless steel · Martensitic transformation

Introduction

Hydrogen effects in metastable austenitic stainless steels are often explained by the low austenite stability of the investigated steel and thus to the amount of α' -martensite formed upon straining, see e.g., [1]. Providing this sole root cause is at least misleading since martensitic transformation is neither necessary nor sufficient to explain hydrogen effects in austenitic stainless steels [2]. However, the formation of a second phase upon straining results in local strain incompatibilities within the microstructure which can result in hydrogen assisted crack initiation [3]. The tensile test results of 10 commercial austenitic stainless steels investigated here were previously published in [4]. The different chemical compositions of the steels resulted in different austenite stabilities and thus, to different α' -martensite fractions on the fracture surfaces. The motivation of this study was to quantitatively measure the α' -martensite content directly on the fracture

surface by XRD and to correlate the result with the loss of tensile reduction in area when tensile tested in gaseous hydrogen to further investigate the role of martensitic transformation directly at the fracture location.

Experimental

The experimental details of the XRD measurements are described in [5] and are only briefly repeated here. XRD measurements were performed on a renewed Siemens D5000 with a DiffracPlus Software from Bruker AXS. The measurements were performed with Cr-K α -radiation with a wavelength of $\lambda = 0.22897$ nm and with 30 kV voltage and 30 mA current in the 2 theta range from 10° to 165° with steps of 0.01°. The penetration depth of the Cr-K α -radiation into the material was about 3–4 μ m, which represents the phase distribution at fracture. For each measurement, the martensite content was calculated according to the procedure outlined in [5]. The advantage of the XRD method is that it works not only on carefully prepared surfaces but also on curved surfaces as well as on rough fracture surfaces.

The chemical compositions, tensile test parameters and tensile test results of the 10 commercial austenitic stainless steels investigated here were previously published in [4]. All steels were tensile tested in helium and hydrogen gas, both at

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Table 1 Results of the XRD measurements on the fracture surfaces of 10 tensile tested austenitic stainless steels, Ni content and reduction in area (RA) from [4]

Steel grade	Equivalent grade	Test atmosphere	α {110}	α {200}	α {211}	γ {111}	γ {200}	γ {220}	α' -martensite vol %	γ -austenite vol %	Ni content %	RA %
X2CrNi-MoN18-14-3	316LN	He	7.66	0.00	0.00	126.15	63.49	159.89	0.94	99.06	13.2	82
X2CrNi-MoN18-14-3	316LN	H ₂	9.36	0.00	1.47	149.06	52.97	141.79	1.38	98.62	13.2	81
X10CrNi-iNb18-9 (A)	347	He	81.58	9.57	28.31	106.49	46.75	131.42	16.37	83.63	11.3	63
X10CrNi-iNb18-9 (A)	347	H ₂	61.69	4.89	16.69	122.37	49.08	120.87	11.83	88.17	11.3	59
X10CrNiTi18-9 (C)	321	He	41.26	0.00	4.36	160.48	49.75	149.88	5.37	94.63	11.5	74
X10CrNiTi18-9 (C)	321	H ₂	38.26	0.17	3.35	147.60	47.67	124.32	5.64	94.36	11.5	65
X2CrNi-iMo17-13-2	TP316NG	He	46.26	4.05	7.10	127.10	50.21	113.90	8.77	91.23	10.4	85
X2CrNi-iMo17-13-2	TP316NG	H ₂	37.08	3.88	8.49	106.49	46.32	129.24	7.61	92.39	10.4	72
X2CrNi19-11	304L	He	50.35	3.38	14.97	120.66	58.96	121.89	9.54	90.46	11.2	73
X2CrNi19-11	304L	H ₂	38.15	5.94	9.73	101.55	57.43	138.32	7.98	92.02	11.2	61
X2CrNi18-10	TP304L	He	62.88	6.43	15.42	134.37	49.94	116.73	12.13	87.87	11.0	83
X2CrNi18-10	TP304L	H ₂	52.91	10.11	22.96	91.97	51.80	116.31	13.78	86.22	11.0	64
X2CrNi18-9	304	He	38.15	0.78	5.79	117.17	51.91	141.37	5.95	94.05	9.9	81
X2CrNi18-9	304	H ₂	50.93	0.00	6.68	169.44	43.11	129.26	7.21	92.79	9.9	61
X6CrNi-iNb18-10 (B)	347	He	63.22	9.13	19.30	147.93	49.25	91.42	14.34	85.66	10.6	60
X6CrNi-iNb18-10 (B)	347	H ₂	87.20	17.09	45.24	94.58	34.39	100.25	24.30	75.70	10.6	38
X10CrNiTi18-9 (D)	321	He	270.20	69.56	184.71	42.50	14.28	41.05	73.08	26.92	9.3	68
X10CrNiTi18-9 (D)	321	H ₂	186.11	20.84	57.72	135.89	39.01	74.08	35.79	64.21	9.3	39
X2CrNi18-10	304LN	He	95.47	16.55	48.33	86.64	44.05	133.54	21.90	78.10	9.6	77
X2CrNi18-10	304LN	H ₂	46.18	3.26	11.07	132.82	59.86	114.68	8.59	91.41	9.6	44

Fig. 1 Representative XRD patterns from the center of the fracture surfaces of steels showing **a** negligible martensitic transformation and **b** severe martensitic transformation

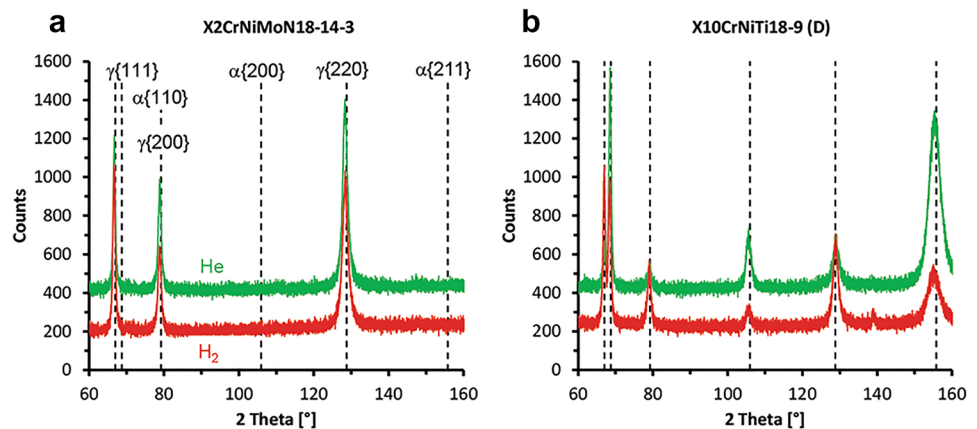
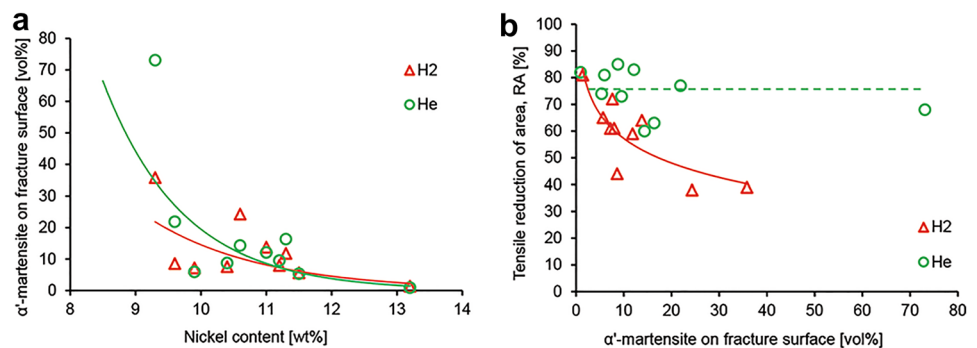


Fig. 2 **a** α' -martensite on the fracture surface as a function of Ni-content and **b** tensile reduction of area as a function of α' -martensite on the fracture for the 10 austenitic steels tensile tested in [4]



a pressure of 9 MPa to investigate the loss of tensile ductility due to gaseous hydrogen. The designation of the steels in this publication is identical to those used in [4] to ensure an easy comparison between the two publications.

Results, Discussion and Conclusions

The detailed XRD results and the calculated martensite fractions on the fracture surfaces of the 10 commercial austenitic stainless steels are summarized in Table 1 together with some tensile data from [4]. Two representative XRD patterns are shown in Fig. 1a, b. As described in [6], the volume fraction of each phase can be calculated by the x-ray intensities from the respective XRD peaks. It is shown in Table 1 and Fig. 1 that α' -martensite and γ -austenite were clearly detected but ϵ -martensite was not detected in either of the steels. It can be further seen from Table 1 and Fig. 1 that the intensities of the α' -martensite and γ -austenite peaks depend on the chemical composition of the steels.

It is well established that nickel is the main austenite stabilizing alloying element in austenitic stainless steels. Figure 2a shows the calculated α' -martensite fractions as a function of the nickel content. The general trend is that α' -martensite content decreases with increasing nickel content for specimens tested in both, He and H_2 atmospheres.

The data in Fig. 2a scatter significantly. However, the scatter could not be reduced by plotting the results as a function of well-known empirical nickel equivalents [1] [7] including M_{d30} temperatures [8] as well as stacking fault energies [9]. In Fig. 2a, the α' -martensite fractions appear to be independent of the test atmosphere for nickel contents higher than about 11 wt%. for lower nickel contents the α' -martensite fractions are lower in H_2 atmosphere compared to He. For a given temperature the amount of transformed α' -martensite mainly depends on the local strain [8]. That is, it appears from these results that the local strain in front of a growing crack is lower in specimens tested in H_2 compared to He, especially for low nickel contents. If this observation complies with the Hydrogen Enhanced Localized Plasticity (HELP) mechanism which is established for austenitic stainless steels, is subject to further investigations.

Figure 2b shows the tensile reduction in area (RA) as a function of the α' -martensite fraction. While RA in He is almost independent of the α' -martensite fraction up to α' -martensite fractions of about 75%, RA in H_2 decreases with increasing α' -martensite fraction from about 80% at about 1% α' -martensite fraction to about 40% at about 40% α' -martensite fraction. The scatter of the test results in H_2 (and also in He) is high which means that the involved fracture mechanisms are complex and cannot be attributed to martensitic transformation alone, as described in detail in [3].

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