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Article

Cr-Si Alloys with Very Low Impurity Levels Prepared by Optical Floating Zone Technique

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Abstract: The optical floating zone technique was utilized to purify chromium and a single-phase chromium-silicon alloy in this work. The impurity content (carbon, nitrogen and oxygen) can be reduced by decreasing the withdrawal speed of samples during the zone refining process and coarsening of grains was also observed. The effect of the impurities on mechanical property was determined by hardness measurements at room temperature, and the hardness of both, chromium and the chromium-silicon alloy were decreased with lower concentrations of nitrogen and oxygen. In contrast, brittle features were observed in samples prepared by arc melting process with higher concentrations of impurities.

Keywords: impurities; Cr-Si alloys; optical floating zone technique; hardness; low temperature brittleness

1. Introduction

Chromium (Cr) – based alloys show good high-temperature properties and are therefore currently investigated intensively [1–9]. However, brittleness at low to moderate temperatures [9–14] often prevent their application. Studies show that carbon (C), nitrogen (N) and oxygen (O) promote brittleness of Cr-based alloys [15–19]. In previous investigations, Cr-based alloys were fabricated using arc melting processes, additive manufacturing or investment casting. These manufacturing processes are not reported to lower impurity levels. Here, chemical and physical interactions between the melt and the mold material or a ceramic crucible respectively can occur, as Pelchen et al. show in their work [20]. The optical floating zone (OFZ) technique, described in detail in the work of Liu et al. [21] and illustrated in Figure 1, is suitable for producing high-purity materials because it enables remelting without a crucible and promotes outward diffusion of impurities [22].

In this work, Cr-based alloys are produced by OFZ to exploit its purification potential. Concentrations of C, N, and O are determined by glow discharge optical emission spectroscopy (GDOES). Martens hardness, also known as universal hardness, is measured to evaluate strength. To evaluate the brittle behavior of the specimens, crack formation on Vickers indentations is evaluated.

2. Materials and Methods

Pure Cr (Plansee SE, purity 99.95%) and pure Si (HMW Hauner, purity 99.999%) are arc melted and solidified in Argon (Ar) atmosphere into cuboid shaped ingots with nominal compositions of 100 at. % Cr and 98 at. % Cr and 2 at. % Si respectively. For OFZ, cylindrical bares with a diameter of 9 mm and a length of 110 mm are prepared from the arc melted ingots by electric discharge machining

(EDM). The bares are further divided with a length ratio of 4:7, the shorter bares thereafter used as so-called seed rods and the longer bares as so-called feed rods in the OFZ. An OFZ furnace, partially shown in Figure 1 b) (FZ-T-12,000-X-VII-VPO-PC from Crystal Systems Corporation, Japan) with four xenon lamps (total power 12 kW) is used to produce nine specimens (Table 1) with two different alloy compositions. A high-pressure quartz glass tube surrounding the seed- and feed rods enables the use of Ar gas in the quality class 6.0 (purity 99.9999%).

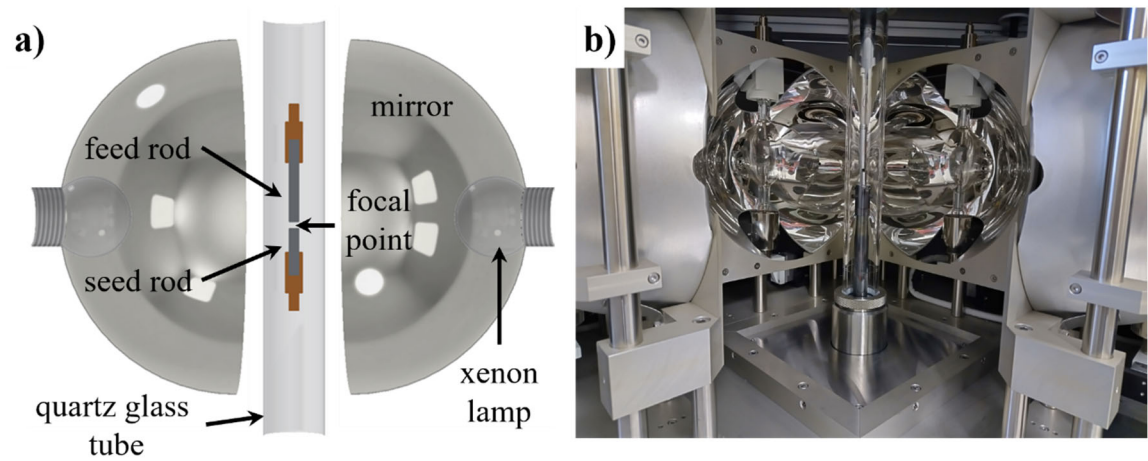


Figure 1. a) schematic drawing of the essential components of an OFZ furnace. Seed- and feed rods are mounted inside a quartz glass tube which can be evacuated or filled with Ar gas. b) View inside the furnace chamber of the OFZ device used for this work. Heat input into the specimens is provided by four xenon lamps with total power of 12 kW.

Table 1 provides an overview of the specimens created in the OFZ and the associated process parameters. The withdrawal speed given in Table 1 is often referred to as growth rate in literature, i.e. the speed at which the seed- and feed rod are moved vertically through the focal point in the OFZ furnace. Further details to the OFZ are described by Liu et al. [21].

Table 1. Specimens processed by OFZ and associated process parameters. All specimen processed in high pressure argon atmosphere of 0.7 MPa except for Cr98Si2_20LP, which is processed in argon under ambient pressure of 0.1MPa.

specimen	nominal composition (in at. %)		withdrawal speed (in mm·h ⁻¹)	Ar pressure (in MPa)	Ar gas flow (in l/min)
	Cr	Si			
Cr100_10	100.0	0.0	10	0.7	2.0
Cr100_20			20		
Cr100_30			30		
Cr100_40			40		
Cr98Si2_20LP	98.0	2.0	20	0.1	
Cr98Si2_10				0.7	
Cr98Si2_20			30		
Cr98Si2_30					
Cr98Si2_40					

After OFZ, specimens are cut lengthwise using EDM so that plane-parallel surfaces are created along the longitudinal axis (Figure 2 c) and d)). Both surfaces are ground stepwise from a grain size of 320 to 1000 on a wet grinding machine and diamond sandpaper and then polished with an aqueous SiO₂ suspension. For electron backscatter diffraction (EBSD) analysis specimens are vibration polished for 24 hours with a 0.06 µm SiO₂ suspension and then ultrasonically cleaned in bath with water and ethanol.

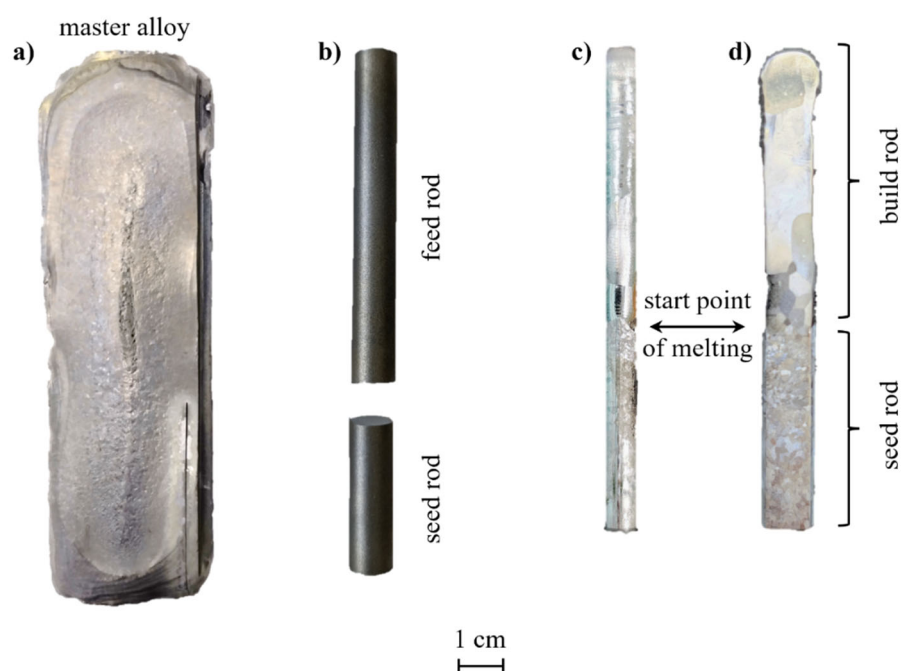


Figure 2. a) Cr₉Si₂ (in at.) master alloy ingot produced by arc melting. b) seed- and feed rod prepared out of the master alloy ingot by EDM. c) side- and d) front view of the specimen produced by OFZ. The build rod represents the remelted volume of the feed rod.

Martens hardness measurements are carried out at room temperature using a Fischerscope® HM 2000. 200 measurement points of the Martens hardness with a test force of 300 mN are recorded along the longitudinal axis of every specimen. Vickers hardness measurements (Matsuzawa VMT-X S3) with a testing force of 98.1 N (= HV10) are carried out on all OFZ processed specimens. The Vickers indentations are investigated for cracks with scanning electron microscopy (SEM).

Microstructures are examined using optical microscopy (OM, Zeiss® Axioplan 2) and SEM, (Zeiss® Gemini 300, Zeiss® Sigma 300 VP and Zeiss® 1540 EsB Cross Beam). Backscattered electron images (BSE) are taken of all specimens. Element distribution maps are recorded by energy dispersive spectroscopy (EDS). Grain sizes are determined from electron backscatter diffraction (EBSD) images. If grains with a diameter larger than 500 µm occur, the grain size is in addition determined by macro etching and OM examination. The chemical composition along the longitudinal specimen axis is measured by µXRF (EDAX® Orbis PC) with a measuring spot of 30 µm in diameter. Phase determination is carried out by X-ray diffraction (XRD) using a Bruker® D8 Discovery.

Carbon (C), nitrogen (N) and oxygen (O) impurity contents are determined using glow discharge optical emission spectroscopy (GDOES, Spectruma® GDA-Alpha). Integral impurity contents of pure Cr from Plansee, pure Cr from EVOCHEM and arc melted Cr₉Si₂ (in at. %) are measured by inductively coupled plasma optical emission spectroscopy (ICP-OES, LECO ONH 836) to evaluate proprietary GDOES standards. Impurity levels of these standards are given in Table 4 and marked with an asterisk *

3. Results

Figure 3 shows a sliced Cr₉₈Si₂ (in at.%) specimen after OFZ, analyzed by SEM, EBSD and EDS. Within the build rod volume, elongated grains are observed (Figure 3 a). The grain length is up to 41 mm. The grain size is additionally determined by macro etching and OM investigations. Figure 3 b) shows the BSE image with corresponding EDS mappings for Cr and Si on the Cr₉₈Si₂_20 specimen as an example for all EDS investigations. EDS mappings verify single-phase microstructure of all Cr₉₈Si₂ specimens.

Table 1 shows the measured length and number of grains in the build rod of the OFZ specimens. Large grains elongated in the withdrawal direction are observed for all specimens, as shown in Figure 3. For the pure Cr specimens, the measured grain length is slightly shorter compared to the Cr₉₈Si₂ specimens.

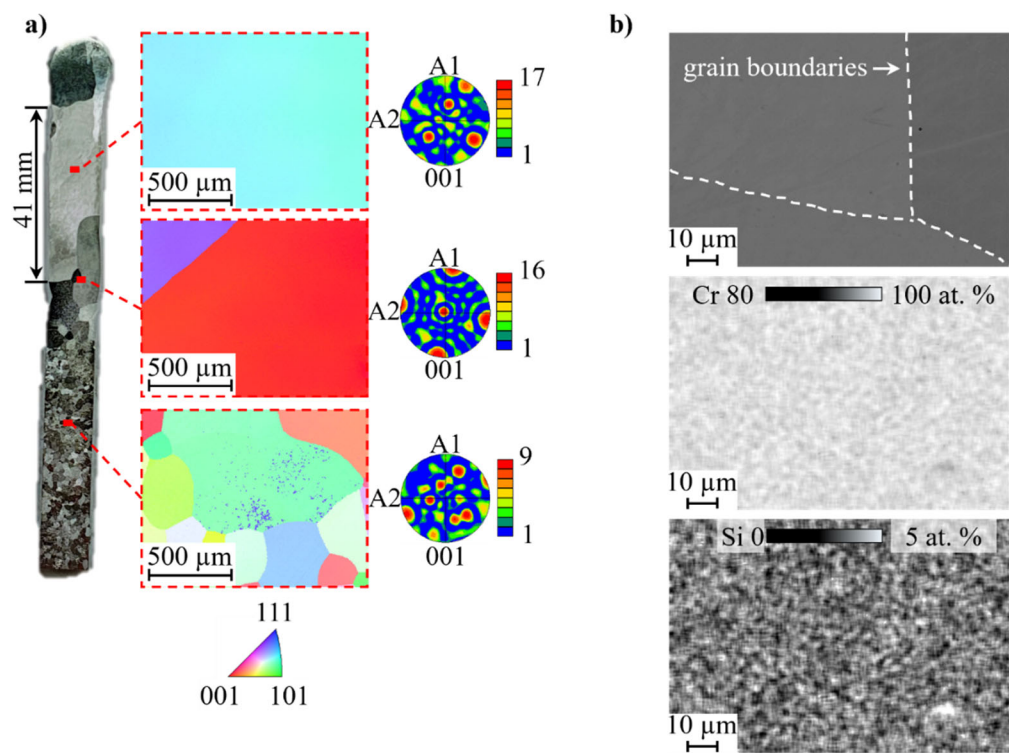


Figure 3. a) EBSD mappings at three different locations on the front surface of the Cr₉₈Si₂_20 specimen with corresponding pole figures. A 41 mm long grain appears in the upper section of the specimen. b) BSE image with EDS mappings for Cr and Si proving the single-phase microstructure of the Cr₉₈Si₂ (in at.%) specimen.

Table 2. Length of the largest grain and number of grains within the build rod of the OFZ specimens.

specimen	nominal composition (in at.%)		length of largest grain (in mm)	number of grains within the build rod
	Cr	Si		
Cr100_10	100.0	0.0	> 30	12
Cr100_20			> 20	16
Cr100_30				17
Cr100_40			9.8	> 30
Cr98Si2_20LP	98.0	2.0	0.6	> 50
Cr98Si2_10			> 40	11

Cr98Si2_20			> 20	12
Cr98Si2_30				22
Cr98Si2_40				26

Table 3 shows the results of the μ XRF measurements of all specimens containing Si. For every specimen, the mean element concentration is calculated from 20 measurements along the longitudinal axis. The alloy compositions do not deviate more than 0.3 at. % from the nominal compositions. OM investigations reveal porosities below 0.5 % for all Cr-Si specimens. Porosity of pure Cr specimens is below 0.25 %

Table 3. Results of the μ XRF measurements and pore fraction analysis by OM on the specimens with Si content.

specimen	nominal composition (in at. %)		μ XRF results (in at. %)		porosity (measured at the surface) (in %)
	Cr	Si	Cr	Si	
Cr100_10	100.0	0.0	100.0 ± 0.0	0.0 ± 0.0	0.15
Cr100_20					0.20
Cr100_30					
Cr100_40					0.25
Cr, pure from Plansee®					0.85
Cr, pure from EVOCHEM®					0.80
Cr98Si2, arc melted	98.0	2.0			0.70
Cr98Si2_20LP			97.6 ± 0.1	2.4 ± 0.1	0.30
Cr98Si2_10			97.8 ± 0.1	2.2 ± 0.1	0.20
Cr98Si2_20			97.9 ± 0.2	2.1 ± 0.2	0.35
Cr98Si2_30			97.7 ± 0.2	2.3 ± 0.2	0.40
Cr98Si2_40			98.1 ± 0.2	1.9 ± 0.2	

XRD patterns reveal a lattice constant of $a_{Cr100}^{100} = 288.3 \text{ pm}$ for the pure Cr specimens and a lattice constant of $a_{Cr98Si2}^{100} = 288.1 \text{ pm}$ for the Cr₉₈Si₂ (in at.%) specimens prepared by OFZ respectively. An influence of the withdrawal speed on the lattice constant is not observed.

Table 4 lists C, N and O levels measured by GDOES. **Table 4** shows an almost constant concentration for C for all specimens produced by OFZ. The concentration for N varies considerably between the specimens. The specimens without Si content have the lowest N content, while the specimens that are not processed with OFZ show significantly higher N concentrations. This also applies to the O contents. With increasing withdrawal speed, an increase in the impurities N and O can be observed for both alloys.

Table 4. Impurities measured by GDOES and of proprietary standard specimens marked with an asterisk *.

specimen	withdrawal speed (in mm·h ⁻¹)	C (in ppm)	N (in ppm)	O (in ppm)
Cr100_10	10	10 ±2	32 ±4	144 ±5
Cr100_20	20	11 ±2	40 ±3	151 ±5
Cr100_30	30	13 ±2	44 ±5	150 ±5
Cr100_40	40	12 ±2	118 ±7	259 ±5

Cr98Si2_10	10	12 ±3	66 ±9	210 ±8
Cr98Si2_20LP	20	12 ±2	82 ±7	211 ±7
Cr98Si2_20		12 ±2	76 ±6	207 ±6
Cr98Si2_30	30	13 ±2	83 ±6	213 ±6
Cr98Si2_40	40	12 ±2	114 ±9	244 ±6
Cr, pure from Plansee®*	--	12 ±2	144 ±5	267 ±5
Cr, pure from Plansee®*		20 ±10	1163 ±50	140 ±50
Cr98Si2, arc melted *		12 ±2	209 ±9	315 ±7

Figure 4 shows the Martens hardness results. For the measured values in **Figure 4** a) and b), the hardness values averaged across all grains are given for each specimen, with scatter bars indicating the deviation. **Figure 4** a) shows the pure Cr specimens. The hardness increases with increasing impurity concentration. The starting material (Cr Plansee®) shows the highest hardness at the highest impurity content. **Figure 4** b) shows an increase in Martens hardness with increasing impurities. The arc melted master alloy shows the highest hardness and the highest content of C, N and O. For both alloys in **Figure 4** a) and b), a grain size effect is ruled out for OFZ specimens because of similar grain sizes. Since the OFZ specimens have very large grains, a grain orientation dependent hardness measurement is possible. **Figure 4** c) and d) show increased Martens hardness in grains where the indentation is perpendicular to the (001) plane of the grain, whereas the hardness perpendicular to the (111) plane is reduced. This observation applies to both alloys.

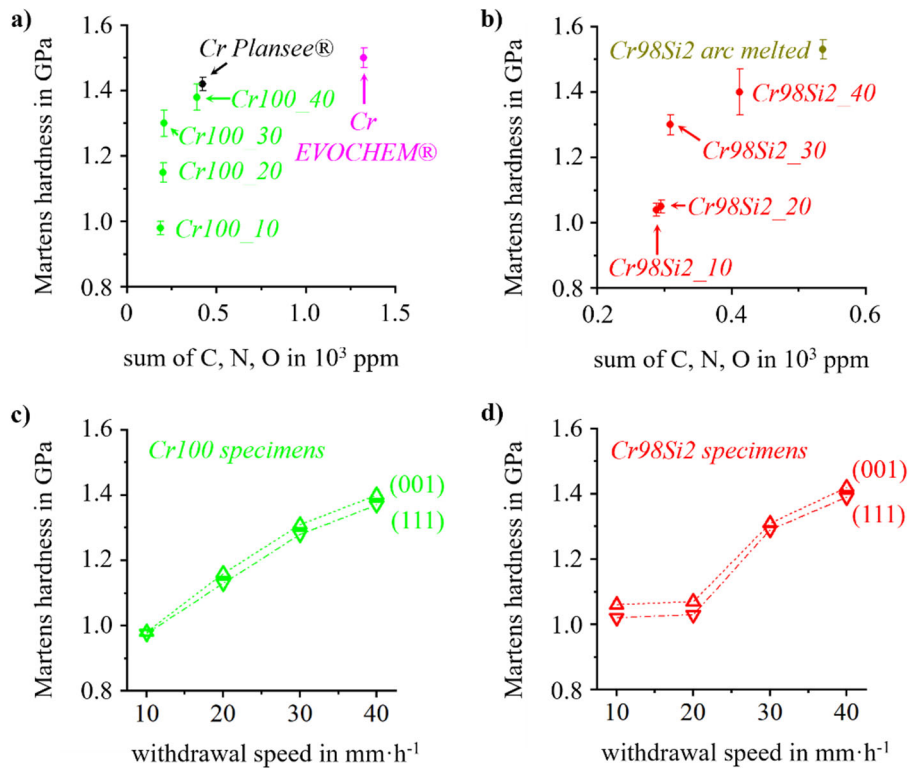


Figure 4. a) measured Martens hardness over the sum of the impurities C, N and O for all four pure Cr OFZ specimens. As reference, the starting material (Cr Plansee®) and Cr with increased N concentration (Cr EVOCHEM®) are given. b) Martens hardness over the sum of C, N and O for Cr₉₈Si₂ (in at.%) specimens prepared by OFZ. The arc melted master alloy showing increased hardness and impurities contents. c) and d)

show increased Martens hardness in (001) direction for Cr₁₀₀ and Cr₉₈Si₂ respectively. The scatter of the measured results is lower than the symbol size.

All Vickers indentations on OFZ specimens are examined for crack formation using SEM. At a resolution of 0.1 μm , no crack formation is observed in any of the OFZ specimens. **Figure 5** a) and b) show two indentations on the OFZ specimens with the highest impurity content (withdrawal speed = 40 $\text{mm}\cdot\text{h}^{-1}$). Vickers indentations are also performed on the starting materials. Pure Cr from Plansee® shows no crack formation (**Figure 5** c), whereas the arc-melted Cr₉₈Si₂ specimen shows crack formation in the corners of the indentations (**Figure 5** d).

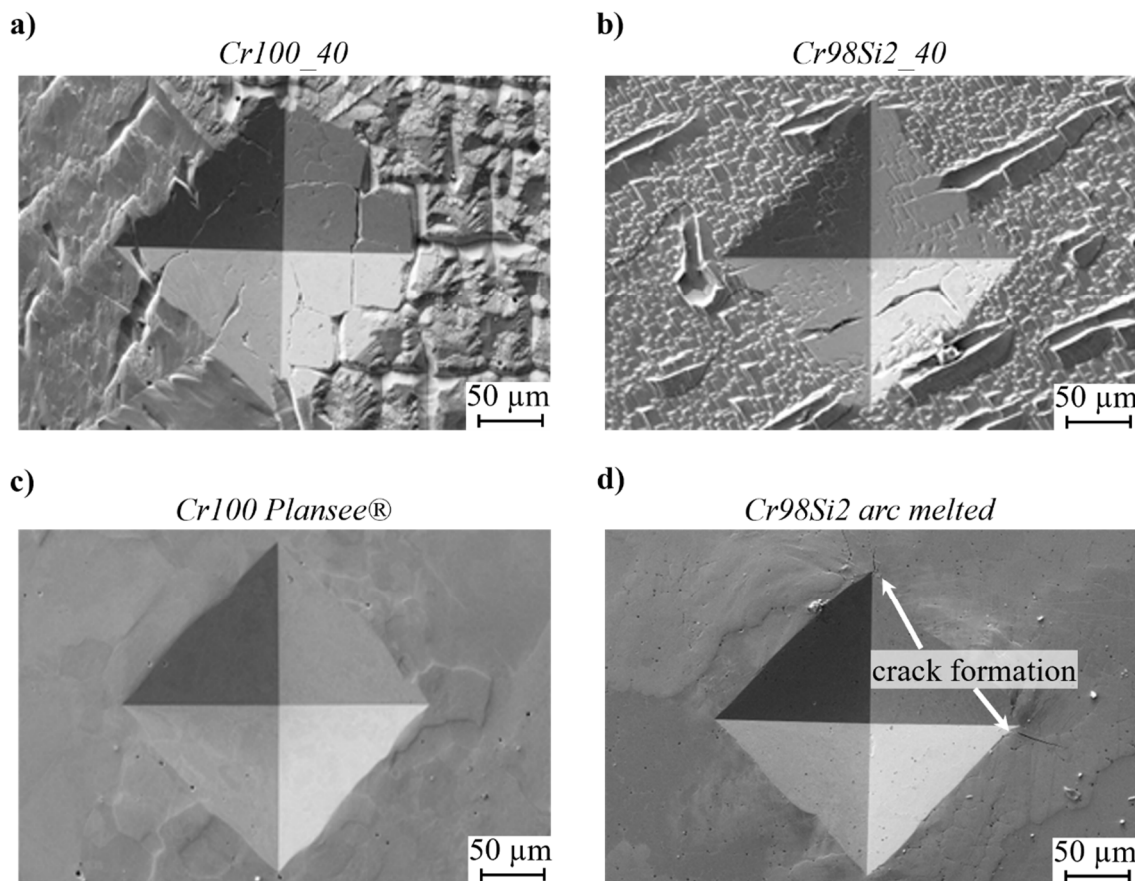


Figure 5. a) and b) show the Vickers indentations (HV10) on OFZ specimens produced with a withdrawal rate of 40 $\text{mm}\cdot\text{h}^{-1}$ for Cr₁₀₀ and Cr₉₈Si₂ (in at.%) after macro etching respectively. No cracks in the edges of the indentations are observed for all OFZ specimens. c) Vickers indentation on the polished surface of the Cr starting material. No cracks are observed. d) Vickers indentation on the polished surface of the Cr₉₈Si₂ (in at.) master alloy showing crack formation at two edges.

4. Discussion

Technically pure Cr and single-phase solid solution dilute Cr-Si alloys both have body centered cubic (bcc) crystal structure and therefore exhibit a brittle to ductile transition [9,23]. The brittle to ductile transition temperatures of these alloys are generally above room temperature [24,25]. In 1935, Kroll [14] showed that pure Cr has ductile properties at room temperature, hence a brittle to ductile transition temperature below room temperature, when the oxygen content is reduced to a technically feasible minimum by multi-stage reduction. Edwards et. al. [26] investigated the insertion of the impurities hydrogen (H), N, O, sulfur (S), phosphorus (P) and other elements during the electrolytic extraction of pure Cr from chromite and determined that from a purity of 99.98 at. % on Cr exhibits ductile, hence plastic deformation at room temperature and above. They also showed that impurities

accumulate preferentially at grain boundaries and conclude that pure Cr should have a coarse-grained microstructure to reduce the risk of impurities entering during exposure to higher temperatures [26]. Pan et al. [27] too found that impurities in refractory metals tend to accumulate at grain boundaries. They used computer simulations to determine the effects of H, O, Si, P and S on the brittle to ductile transition of tungsten [27]. A low N content is crucial for Cr-Si alloys, since previous studies have shown that N leads to embrittlement of Cr-Si alloys [28].

In this study the impurities N and O are again identified to be crucial for the brittle to ductile transition of pure Cr and dilute Cr-Si alloys. The melting metallurgical process OFZ turned out to purify Cr and dilute Cr-Si alloys to an extent that the brittle to ductile transitions take place below room temperature. The lower the OFZ withdrawal speed, the lower the impurities levels in the processed alloys. In addition, OFZ processed alloys have low porosities and homogeneous microstructures. OFZ generates coarse elongated grains, which is not possible with other melting metallurgical processes such as arc melting or investment casting [29,30]. The melting metallurgical OFZ process is an alternative to the chemical processes of multi-stage reduction [14] and electrolytic extraction [26] of the elemental alloy components to obtain high purity Cr and dilute Cr-Si alloys.

5. Conclusions

Cr and Cr-Si alloys can be purified by OFZ. In particular, the OFZ process results in very low N and O levels. The slower the OFZ withdrawal speed, the lower the impurities levels. Low N and O levels together with grain coarsening through OFZ processing affected the deformation behavior of Cr and Cr-Si alloys. By OFZ purified and coarsened Cr and Cr-Si alloys have decreased hardnesses, but do not show brittle fracture at room temperature. Hence these purified alloys have brittle to ductile transition temperatures below room temperature.

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Abbreviations

The following abbreviations are used in this manuscript:

EBSD	electron backscattered diffraction
EDM	electric discharge machining
EDS	energy dispersive spectroscopy
GDOES	glow discharge optical emission spectroscopy
ICP-OES	inductively coupled plasma optical emission spectroscopy
OFZ	optical floating zone
OM	optical microscopy
ppm	parts per million
SEM	scanning electron microscopy
XRD	X-ray diffraction
μXRF	Micro- x-ray fluorescence spectroscopy

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