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Tensile properties of EC316LN irradiated in SINQ to 20 dpa

Y. Dai *, G.W. Egeland, B. Long

Spallation Neutron Source Division, Paul Scherrer Institut, CH-5232 Villigen PSI, Switzerland

Abstract

The wide application of 316-type austenitic stainless steels in existing spallation targets requires a comprehensive understanding of their behavior in spallation irradiation environments. In the present study, EC316LN specimens were irradiated in SINQ targets to doses between 3 and 17.3 dpa at temperatures between about 80 °C and 390 °C. Tensile tests were conducted at room and irradiation temperatures. The results demonstrate that the irradiation induced significant hardening and embrittlement in the specimens. The irradiation hardening and embrittlement effects show a trend of saturation at doses above about 10 dpa. Although the ductility was greatly reduced, all specimens broke with strong necking, which indicates a ductile fracture mode.

1. Introduction

Austenitic stainless steels 316- or 304-types (SS 316 or SS 304) are widely applied in nuclear power plants and also extensively used in spallation targets. The neutron irradiation data show that at temperatures above about 400 °C these austenitic steels have low resistance to irradiation induced swelling and creep. However, at lower temperatures, they may have much better resistance to irradiation induced embrittlement effects as compared to ferritic/martensitic (FM) steels. In particular they do not have any problems like a ductile-to-brittle transition temperature shift after irradiation as in the case of FM steels. This leads to austenitic steels being selected for low temperature applications, e.g. in the targets of the existing spallation neutron sources such as ISIS, SINQ and SNS (the English, Swiss and American spallation neutron sources, respectively). Although SS 316 and SS 304 are widely used in spallation targets, the changes of their mechanical properties and microstructure after irradiation in spallation environments still need better understanding, particularly at high dose levels above 10 dpa. In our previous study, we investigated the microstructure of EC316LN and its weld metal irradiated in SINQ Target-3 up to 11 dpa at temperatures up to 350 °C [1]. The results showed that, in addition to small 'black-dot' defects and large Frank loops usually observed after neutron irradiation, small helium bubbles of about 1.2 nm were also observed in specimens irradiated to about 10 dpa at $\geqslant \sim 300$ °C. Similar results were obtained from the observations on the specimens irradiated in STIP-II [2]. In the present work, tensile tests were performed on the specimens from the same irradiation experiments aiming at understanding the changes of tensile properties.

2. Experimental

2.1. Material and specimens

The EC316LN steel and part of the specimens were supplied by the Oak Ridge National Laboratory (ORNL). The steel was solution annealed at 1050 °C for 30 min. The chemical composition in wt% is: 17.45Cr, 12.2Ni, 2.5Mo, 1.81Mn, 0.024C, 0.39Si, 0.067N, and balance Fe.

Two types of miniature flat tensile specimens, so-called 'S-Tensile' and 'L-Tensile', have been used. The S-type specimens have a gauge section of 0.4 × 1 mm cross-section and 5 mm length. The L-type specimens have the same gauge length of 5 mm, but a larger cross-section of 0.75 × 1.5 mm. The dimensions of the specimens are shown

* Corresponding author. Tel.: +41 56 310 4171; fax: +41 56 310 4529.
E-mail address: yong.dai@psi.ch (Y. Dai).

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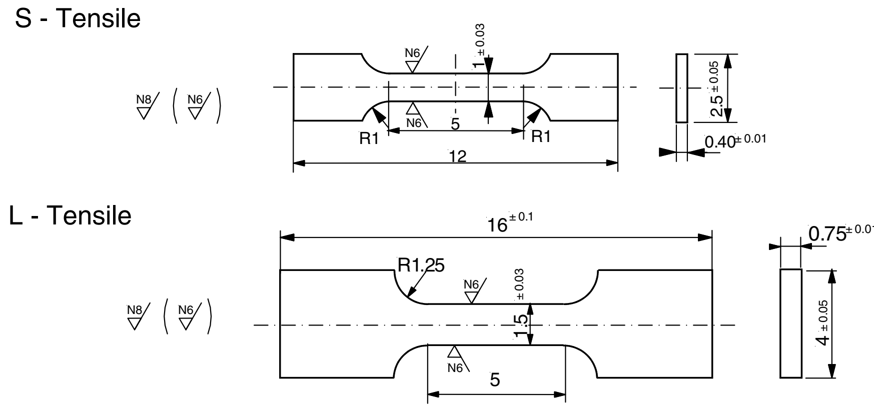

Fig. 1. Sketch showing the dimensions of the S- and L-type of tensile specimens.

in Fig. 1. The specimens were cut with an electro-discharge machine and then finished with mechanical polishing using #1000 sandpaper.

2.2. Irradiation

The specimens tested in this work were included in the first and second experiments of the SINQ Target Irradiation Program (STIP-I and -II) performed in SINQ Target-3 and Target-4 through 1998–1999 and 2000–2001, respectively. Target-3 was irradiated for about 14 months during 1998 and 1999 and a proton charge of 6.8 Ah was received [3]. Target-4 was irradiated for 16 months in 2000 and 2001 and the proton charge was 10.03 Ah [4]. The proton and neutron fluences received by each specimen, the corresponding dpa values and helium and hydrogen concentrations were calculated with the MCNPX code using the profile of the accumulated proton fluence evaluated by gamma-mapping performed on the beam entrance area of the $AlMg_3$ container, as explained in detail in [4]. Calculated neutron and proton fluences were compared to experimental values obtained from gamma spectra measurements of dosimetry foils. The calculated values were found to be in relatively good agreement with experimental data, although slightly (~10%) larger [5]. The helium (He) and hydrogen (H) contents were also calculated using the calculated proton and neutron fluences multiplied the He and H cross-sections which were updated based on the measurement results from an irradiation program at Los Alamos National Laboratory (LANL) [6]. Furthermore, the calculated He concentrations have been corrected based on the gas measurements performed at the Pacific Northwest National Laboratory, USA [5]. The H concentration could not be corrected because the measured H values showed a large difference from the calculated values. The difference depended strongly on irradiation temperature. Those specimens irradiated at >200 °C, where H diffused out of the specimens, showed much lower measured values.

The irradiation temperature was monitored during irradiation using several thermocouples installed in different specimen rods. The irradiation temperature was not constant during the experiment due to the variation of the proton beam current during two years, from about 0.9 mA to 1.25 mA, and frequent beam trips, about 50 times per day. Furthermore, a slight over-focusing of the proton beam caused a high temperature excursion of a few minutes duration in October 1999 in STIP-I, and another one for a period of 22 h in October 2000 in STIP-II. The temperature of each specimen was calculated using the ANSYS code as discussed in detail in [3,4].

Table 1 lists the irradiation parameters of the specimens used in this work: the irradiation dose, He and H concentrations, the average irradiation temperature and the upper- and lower-bound temperatures during normal operation (excluding the beam trips), and the maximum temperature reached during the 22-h abnormal beam focussing of STIP-II. In the table, the calculated H concentrations are just for a reference, which are most probably much greater than the unknown actual values.

2.3. Tensile testing

The tests were conducted on a 2 kN MTS testing machine for the S-type specimens and on a 10 kN Zwick testing machine for the L-type specimens. Both testing machines were equipped with furnaces. The MTS machine was also equipped with a video-extensometer. On the MTS machine the tests were conducted in flowing N_2 gas, and the elongation was measured directly from the gauge section with the video-extensometer. While on the Zwick machine the tests were done in Ar gas and the elongation was taken from cross-head travel with a correction of the compliance of the machine. The same nominal strain rate, 1×10^{-3}/s, was used for all the tests. The tests were divided into two groups according to testing temperature: one was at room temperature (25 °C) and the other at temperatures within the irradiation temperature range of the specimens, which varied from about 100 °C to about 400 °C. The testing temperatures are also listed in Table 1.

The SEM observations of the fracture surface of the SS 316LN specimens have not been performed due to the high activity of the specimens. But some photos were taken with

Table 1
The irradiation dose, calculated He and H levels, testing temperatures and the tensile properties of the specimens

Specimen ID[a]	dpa	He (appm)	H (appm)	$T_{irr.}$ (°C)	T_{test} (°C)	YS (MPa)	UTS (MPa)	STN (%)	TE (%)
JUI01					25	295	588	42	47.4
J22	3	305	1155	84, 77/91	25	703	764	32	37
J12	4	430	1580	117, 107/126	25	780	821	27.3	33.7
J23	5.3	265	2400	154, 142/167	25	878	894	21	25.7
J20	7.6	463	3410	222, 204/240	25	891	934	13.9	19
J16	9.7	742	4425	288, 265/311	25	965	965	9.6	14.5
J10	11.3	931	5190	337, 309/364	25	961	968	14.6	21.3
J14	4	430	1580	117, 107/126	100	662	693	24.4	29
JUI02					150	208	495	33.7	39
J7	5.3	265	2400	157, 144/169	150	706	707	9.4	12.8
JUI03					250	160	445	29	35.3
J18	7.6	463	3410	226, 207/244	250	713	726	3.9	7.8
JUI03					350	158	435	28.5	32.8
J15	9.7	742	4425	292, 269/316	300	749	749	1.1	8.2
J2	11.3	931	5190	341, 314/370	350	710	712	3.9	8.4
A20	17.3	1735	7500	347, 300/391, 477	350	750	755	1	12.3
A32	17.3	1735	7500	347, 300/391, 477	400	743	743	1	9.6

[a] Specimens with ID 'Jxx' are S-type irradiated in Target-3 (STIP-I), while with ID 'Axx' are L-type irradiated in Target-4 (STIP-II).

an optical microscope, which could give an indication of the fracture mode of these specimens.

3. Results and discussion

The results of the tensile tests are presented in Figs. 2 and 3. Fig. 2 shows the engineering tensile stress–strain curves of both irradiated and unirradiated specimens tested at room temperature (RT). With irradiation dose increasing from 3 dpa to 9.7 dpa, the yield stress (YS) and ultimate tensile strength (UTS) increased while the uniform and total elongations decreased. This indicates a general trend of hardening and embrittlement with dose in the low temperature regime. At the highest dose of 11.3 dpa, the hardening did not further increase while the elongation

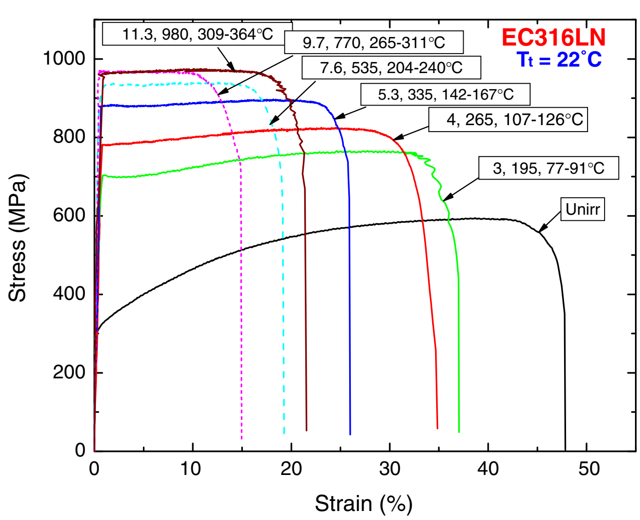

Fig. 2. Tensile stress–strain curves of unirradiated and irradiated EC316LN specimens tested at room temperature. The numbers in the label of a curve indicate: the irradiation dose (in dpa), helium content (in appm) and irradiation temperature range.

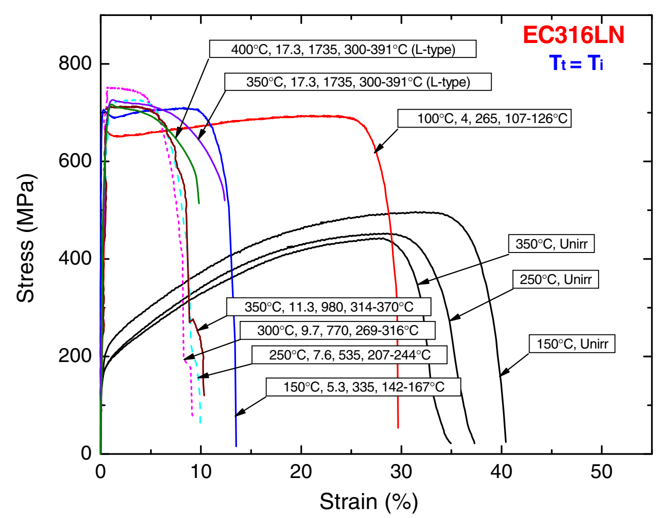

Fig. 3. Tensile stress–strain curves of unirradiated and irradiated EC316LN specimens tested at approximate irradiation temperatures between 100 °C and 400 °C. The numbers in the label of a curve indicate: the testing temperature, irradiation dose (in dpa), helium content (in appm) and irradiation temperature range.

or ductility recovered a little as compared to that of the 9.7 dpa specimen. This might be attributed to the higher irradiation temperature of the 11.3 dpa specimen.

Fig. 3 presents the engineering tensile stress–strain curves of the specimens tested at elevated temperatures ranging from 100 °C to 400 °C. Compared to the results of room temperature tests, it can be seen that the higher testing temperatures had an evident effect on the strength and ductility. For the unirradiated specimens, the strength and ductility decreased with increasing testing temperature in the temperature range. For the irradiated specimens, as the irradiation temperature was increasing with proton flux (or with irradiation dose), the specimens of higher doses were tested at higher temperatures. Similar to the case of unirradiated specimens, the strength and ductility of an irradiated specimen reduced significantly. For example, the yield strength of the 9.7 dpa specimens decreased from about 970 MPa at 25 °C to about 750 MPa at 300 °C, and meanwhile the strain-to-necking (STN) decreased from 9.6% to 1%.

From the results shown in Figs. 2 and 3, one may see that the irradiation hardening seems to saturate at higher doses above ∼10 dpa. Meanwhile the STN drops to ∼1% as prompt necking occurs after yielding. However, the total elongation (TE) remains at a level above 5%.

As SEM observation could not be performed, some pictures were taken on an optical microscope to identify the fracture mode. Fig. 4 illustrates a few representative specimens: (a) the S-type unirradiated specimen tested at 350 °C, (b) the S-type specimen of 9.7 dpa tested at RT, (c) the S-type specimen of 9.7 dpa tested at 300 °C, and (d) the L-type specimen of 17.3 dpa tested at 350 °C. Although the details of the fracture surfaces cannot be seen, the pictures show obviously that all these specimens broke in a ductile fracture mode because strong necking took place before rupturing, even in the most brittle case of the 9.7 dpa specimen tested at 300 °C.

In the STIP irradiation experiments, several austenitic steels such as EC316LN, SS316L, J316F and JPAC were studied in the last few years [7,8]. Fig. 5 presents the irradiation dose dependence of the YS, UTS, STN and TE of these steels tested at room temperature. One can see that the data points of EC316LN, SS316L and JPCA are very close, especially for YS, UTS and TE data. The J316F steel shows less hardening, but not greater rather less STN than the other steels.

Fig. 6 presents the dose dependence of YS, UTS, STN and TE of the four kinds of austenitic steels tested at 100–350 °C. Although both strength and ductility changed evidently when testing temperature increased from 25 °C to e.g. 150 °C, the changes were much less pronounced when the temperature increased from 150 °C to 350 °C. This can be seen from the data points of unirradiated specimens of different steels shown in Figs. 5 and 6. Therefore, it is believed that the difference in strength or ductility among the irradiated specimens is mainly induced by irradiation and less affected by testing temperature (between 100 °C and 350 °C). One important feature demonstrated by this figure is the saturation in both strength and ductility at doses above ∼10 dpa.

Compared to that austenitic steels irradiated at with protons [9] or neutrons [10] at low temperatures (⩽250 °C), the YS and UTS of these steels saturate at about the same level. As for the ductility, the results indi-

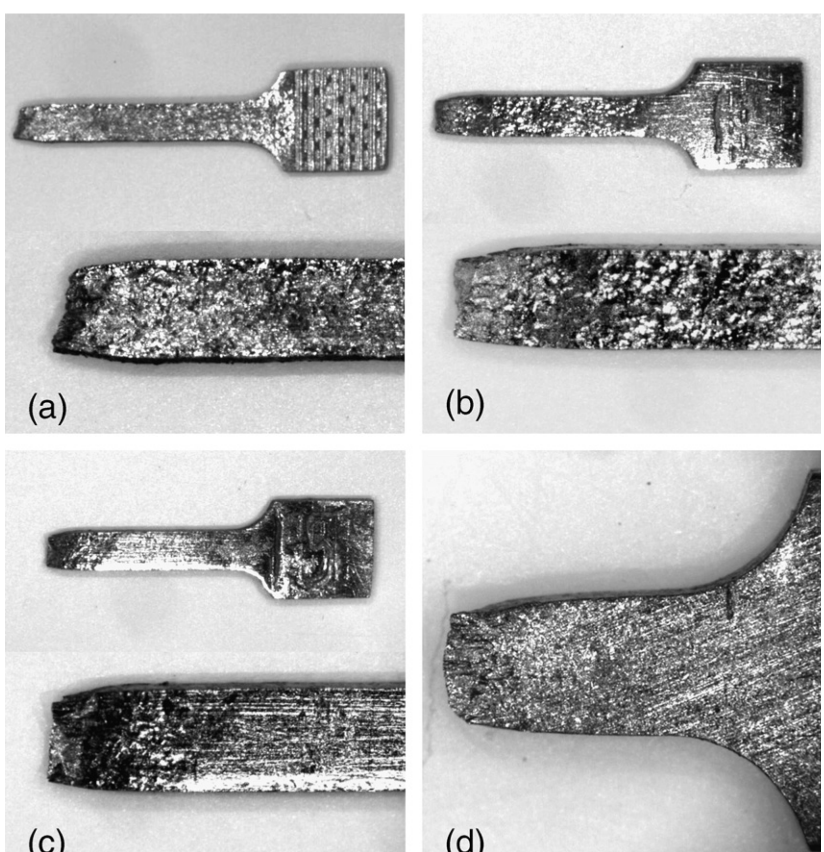

Fig. 4. Pictures of SS 316LN specimens: (a) S-type unirradiated specimen tested at 350 °C, (b) S-type specimen of 9.7 dpa tested at RT, (c) S-type specimen of 9.7 dpa tested at 300 °C and (d) L-type specimen of 17.3 dpa tested at 350 °C.

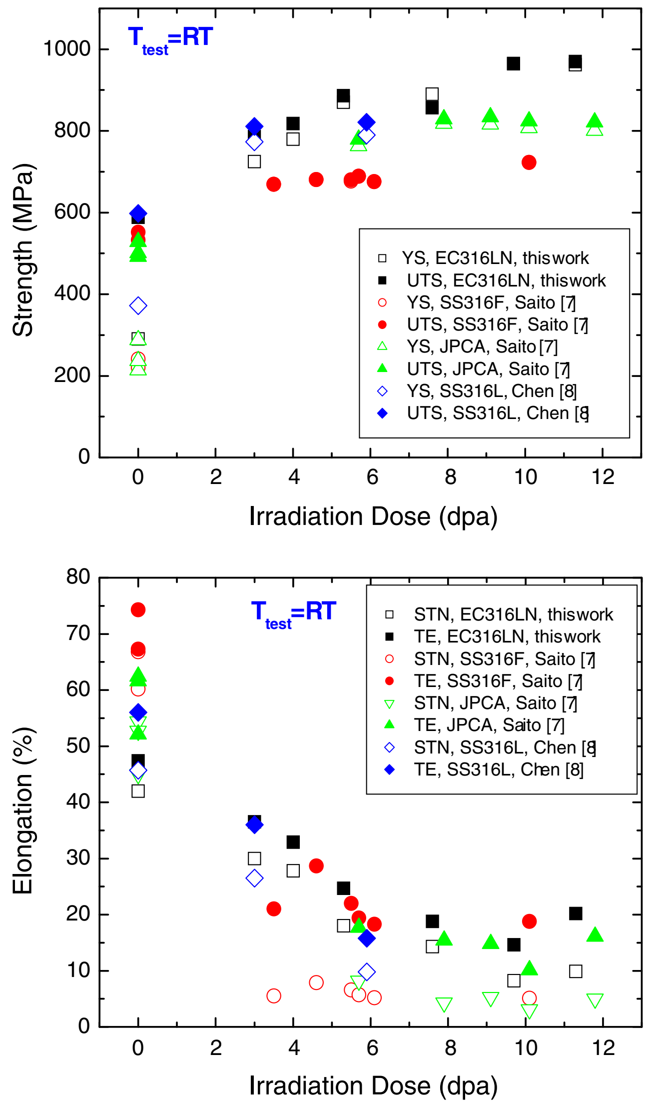

Fig. 5. Irradiation dose dependence of the yield strength (YS), ultimate tensile strength (UTS), strain-to-necking (STN) and total elongation (TE) of austenitic steels irradiated in STIP-I and tested at room temperature.

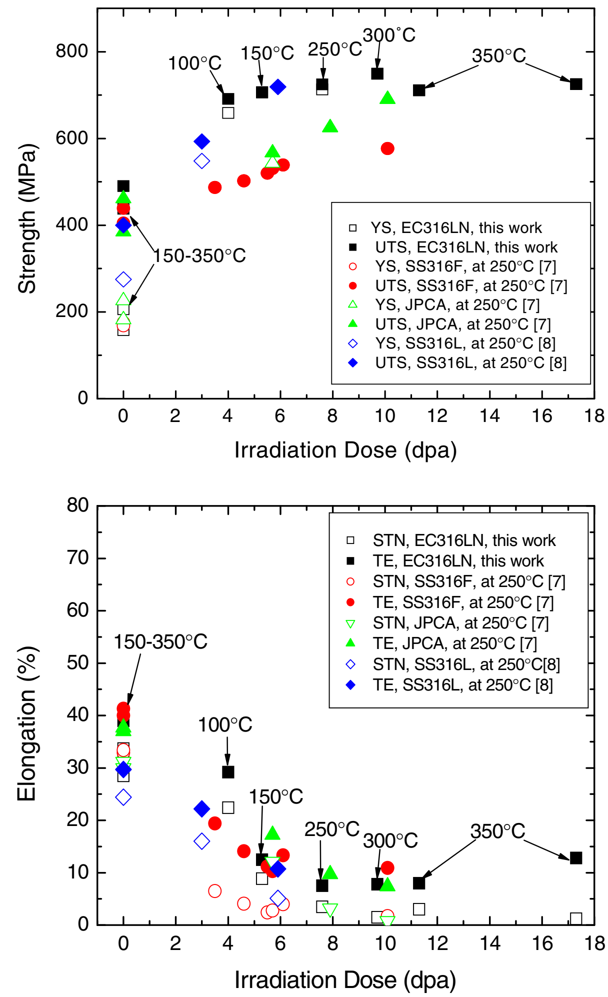

Fig. 6. Irradiation dose dependence of YS, UTS, STN and TE of austenitic steels irradiated in STIP and tested temperatures between 100 °C and 350 °C. The temperature values indicated in the figure are the testing temperatures.

cate that the decrease of the TE stops at about the same dose, though the STN decreases further to essentially <1%, which was observed in other irradiations, too [9,10]. A conclusion drawn from such a comparison is that, as also can be seen from the large database shown in [11], no substantial difference is exhibited in the tensile properties of 316-type steels irradiated in fission reactors and spallation targets in the present dose and temperature ranges, particularly for those specimens tested at ⩾100 °C. This also indicates that He does not introduce a noticeable hardening effect, although He-bubbles were observed in specimens irradiated to high doses at <∼300 °C [1]. This conclusion is in agreement with that obtained from ion-irradiation [12], which shows no additional hardening at <∼5000 appm He. It is believed that the He-bubbles, no matter whether they are visible or not, are mostly combined with defect clusters and such a complex of He-bubbles and defect clusters has more or less the same hardening as that of defect clusters. More detailed investigations on this issue are ongoing.

4. Conclusions

EC316LN specimens were irradiated in STIP-I and -II to doses between 3 dpa and 17.3 dpa at temperatures between about 80 °C and 390 °C. Tensile tests were conducted at RT and irradiation temperatures. The following conclusions can be drawn from the results.

(1) The irradiation in the SINQ targets induces significant hardening and embrittlement in the EC316LN steel in the present irradiation dose and temperature ranges.
(2) The irradiation hardening and embrittlement effects show a trend of saturation at doses above about 10 dpa.
(3) Although the ductility was substantially reduced, all specimens broke with strong necking, which indicates

a ductile fracture mode. Meanwhile, the total elongation remains above 5%.

(4) Compared to published results from other austenitic steels irradiated in STIP-I, the tensile properties of these steels are very similar after irradiation. The present results also agree with published results from high energy proton or neutron irradiation.

(5) He or He-bubbles in the present specimens do not show a noticeable additional hardening effect.

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