



Nanoindentation study on solid solution softening of Fe-rich Fe₂Nb Laves phase by Ni in Fe–Nb–Ni ternary alloys



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ABSTRACT

In an attempt to understand the solid solution softening by Ni of an Fe-rich Fe₂Nb Laves phase that is in equilibrium with γ -Fe, we have examined the defect structure in the Laves phase in Fe–Nb–Ni alloys by transmission electron microscopy, and its hardness as a function of orientation and solute (Ni) content by nanoindentation. The binary Fe-rich Laves phase and the ternary Laves phases with lower Ni content (18%) exhibit a featureless morphology with low dislocation density, whereas the ternary Laves phase with higher Ni content (33%) includes basal planar faults extending to several micrometers, producing a local change in the stacking sequence of the three 3⁶-nets (triple layer) of the C14 structure. The hardness of the binary and the ternary Laves phases with 18% Ni in solution is almost constant and independent of orientation, whereas the ternary Laves phase with 33% Ni in solution exhibits a substantial dependence of hardness on orientation and this dependence appears associated with the ease of basal slips evidenced by slip traces around nanoindents. The relative ease in activating basal slip in the presence of a large amount of Ni in solution is thought to be the dominant contributor to the observed solid solution softening.

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1. Introduction

A common class of intermetallic compounds with the AB₂ stoichiometry and complex crystal structures is Laves phase. This phase occurs as binary compounds, typically over a small range of compositions, with sometimes extensive solubility for a third element, and occasionally, as uniquely ternary compounds in some systems. In some instances however, such as in the binary Fe–Nb system that is the subject of this study, the C14 Fe₂Nb Laves phase can be stable at compositions that are significantly off-stoichiometric. The more common forms are the hexagonal C14 and cubic C15, with a minor form of the hexagonal C36 type [1]. These phases are notoriously brittle at ambient temperature, their brittleness being attributed to the difficulty in moving dislocations under an applied shear stress [1–4]. These compounds are often present in many multiphase alloys, and are believed to have a detrimental effect on the overall mechanical properties,

particularly, ductility and fracture toughness at ambient temperatures [5].

As a part of our on-going effort to develop novel austenitic heat-resistant steels strengthened by thermodynamically stable Laves phase, the phase equilibrium between γ -Fe and Fe₂M Laves phases (C14 structure) has been systematically examined in Fe–M–Ni ternary systems (M: Nb, Ti, Mo) at elevated temperatures [6–10]. Takeyama and coworkers have recently proposed a new austenitic heat-resistant steel, a carbon-free alloy with the composition Fe–20Cr–30Ni–2Nb (at.%) that is strengthened by thermodynamically stable TCP (topologically close-packed) Laves phases and other GCP (geometrically close-packed) phases [7,8,11]. They demonstrated that this steel exhibits superior long-term creep rupture strength due to the Laves phase sufficiently covering grain boundaries [11–13]. Extensive phase diagram studies demonstrated that in the Fe–Nb–Ni ternary system, a homogeneity region of the Fe₂Nb Laves phase with the C14 structure extends towards the equi-Nb concentration direction up to 44 at.% Ni [10]. This indicates that more than two thirds of the Fe sublattice sites in Fe₂Nb can be replaced by Ni atoms. The solute Ni atoms preferentially replace Fe₂ sublattice site in C14 structure, as revealed by XRD

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measurement [14]. An interesting finding in the Fe–Nb–Ni ternary Laves phase is the significant solid solution softening measured in the Fe-rich Laves phase (in equilibrium with γ -Fe). The hardness of Laves phase measured by Vickers indentation is plotted as a function of solute Ni content in Fig. 1. A part of the hardness data and the chemical compositions have been shown elsewhere [6,10]. We note that the hardness of the Fe-rich Laves phase decreases substantially with increasing Ni content, especially beyond 25 at.% Ni and up to about 40 at.% Ni from 9.1 GPa to 4.5 GPa. Whether a similar response is present for the stoichiometric Laves phase is not known as compositions with Ni (substitution for Fe) greater than 20% were not examined. Associated with these measurements is a large scatter in Vickers hardness values. The origin of this scatter is not clear but could be associated with the change in hardness of the Laves phase with orientation (i.e. basal slip). Ni in solid solution may enhance the plastic deformability of the Laves phase by facilitating synchroshear and promoting basal slip [15–17], resulting in solid solution softening. We have recently demonstrated the possibility of plastic deformation by slip in compression at room temperature in single crystal micropillars of a Fe–Nb–Ni ternary Laves phase [18]. Another possible reason for the scatter in the Vickers hardness may be related to the presence of the soft surrounding γ -Fe phase. Thus, the observed hardness change is attributable to different intrinsic and extrinsic factors, making it difficult to isolate solid solution softening as the dominant effect. To further elucidate this effect, in the present study, we have examined the hardness of Fe-rich Fe_2Nb Laves phases (in equilibrium with γ -Fe) by nanoindentation as a function of orientation and Ni content in solid solution in the Laves phase. The observations are then utilized in conjunction with previous results to understand the origin of solid solution softening.

2. Experimental procedure

The alloys examined in this investigation are nominally Fe–15Nb–X alloys where X represents Ni ranging from 0 to 40 at.% (Fe–15Nb–(0, 20, 40)Ni), and hereafter all compositions are given in atomic percent (at.%) unless otherwise noted. These alloys were prepared by arc-melting and each weighed about 30 g. The prepared alloys were cut into pieces and then equilibrated at 1473 K for 240 h, followed by a water quench to room temperature. Fig. 2 shows the isothermal section of the Fe–Nb–Ni ternary phase diagram at 1473 K [10]. Compositions of these alloys fall within the two phase region of γ -Fe and Fe_2Nb Laves phase. The specimens for

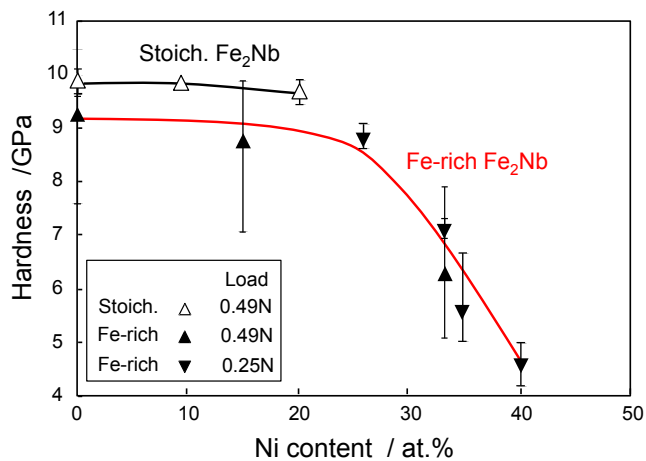


Fig. 1. Change in Vickers hardness of Fe_2Nb Laves phases with stoichiometric and Fe-rich compositions as a function of the Ni content in solution. The hardness was measured using loads of 0.25 N and 0.49 N.

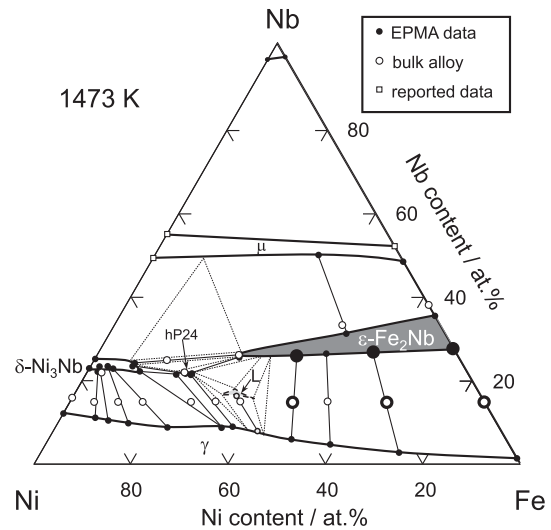


Fig. 2. Isothermal section of the Fe–Nb–Ni ternary phase diagram at 1473 K [10].

optical microscopy and scanning electron microscopy (SEM) were mechanically polished, followed by electropolishing in a Cr-saturated phosphoric acid solution at 35 V, 0.5 A for 30 s at 337 K. The phases and crystallographic grain orientation were analyzed by EBSD (TSL solutions, OIM Data Collection and OIM analysis). Thin discs for transmission electron microscopy (TEM) were cut from the heat treated alloy, mechanically polished, and twin-jet electro-polished in a solution of ethanol with 12 vol.% perchloric acid at 253 K. TEM observation was carried in a JEOL JEM-2100F microscope. High resolution TEM (HRTEM) images of the Fe_2Nb phase with the C14 structure were simulated using a multi-slice method [19].

Nano-indentation was performed with a Berkovich triangular pyramid indenter [20] using a HYSITRON Triboindenter at a maximum load of 40 mN. A minimum of 30 indentation tests were performed for each orientation in each of the three Laves phases at a constant loading rate of 2 mN/s and a holding time of 2 s. All indents on the sample surface were observed by SEM in order to determine whether there were any cracks around the indent. We have excluded all hardness data where the indents were accompanied by microcracks on the sample surface. We note that, these microcracks were observed only in the Fe-rich binary Laves phase in the Fe–15Nb alloy.

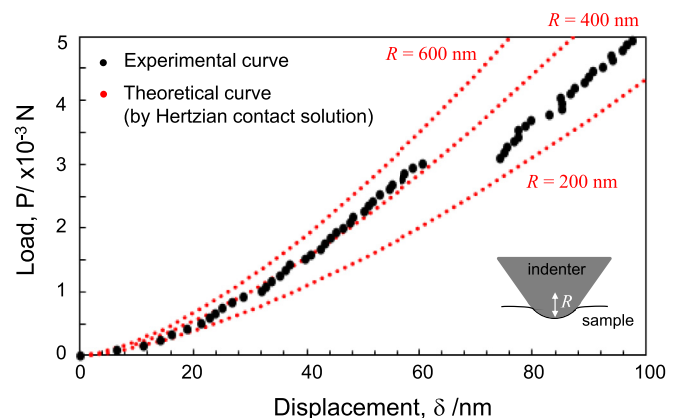


Fig. 3. A representative nanoindentation load–displacement curve for the Laves phase in a binary Fe–15Nb alloy, together with theoretical curves calculated by Hertzian contact solution assuming an indenter tip radius R of 200, 400 and 600 nm.

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