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Hot deformation of a Fe-Mn-Al-C steel susceptible of $\kappa\text{-carbide}$ precipitation



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ABSTRACT

The mechanical properties of Fe-Mn-Al-C steel are significantly enhanced after κ -carbide precipitation via aging; however, most aging treatments are energy demanding because they require relatively high temperatures and extended holding times. This research determined that the precipitation of these carbides can also occur within a few seconds of thermomechanical treatments (TMTs). This behaviour has not been reported post-TMTs for this steel group. Hot compression tests were performed on Fe-21Mn-11Al-1.5C-2Si wt% specimens at test temperatures ranging from 900 °C to 1150 °C and strain rates varying from 0.01 s⁻¹ to 1 s⁻¹. The effects of strain rate and test temperature on dynamic recrystallization behaviour were evaluated. The microstructures were characterized by scanning electron microscope and electron backscatter diffraction. Hardness tests were performed before and after applying processes studied i.e., TMT and aging treatment to determine the change in hardness induced. Particularly, nanoindentation tests were also used to collect indirect evidence about the deformation mechanisms. The load-displacement curves *P*-*h* and (*P*/*h*)-*h* showed the occurrence of several popins and slope changes related to the nucleation of dislocations and strain-induced phase transformations. The occurrence of these phenomena is discussed.

1. Introduction

Steel alloys from the Fe-Mn-Al-C system have garnered significant attention over the last two decades due to their exceptional combination of mechanical properties that show promise for many applications [1]. Steel alloys with elements ranging between 20 and 30 wt% of Mn, 0-12 wt% of Al and 0.6-1.5 wt% of C are particularly attractive due to the combination of properties such as reduced weight (from 1% to ~16%) [2–4], corrosion resistance [5], high strength (ultimate tensile strength (UTS) from ~0.5 GPa to ~2 GPa) [4,6] and high ductility (from $\sim 10\%$ to $\sim 80\%$) [4,7]. This useful combination of high strength and high ductility relies on special deformation mechanisms such as transformation-induced plasticity (TRIP), twinning-induced plasticity (TWIP), microband-induced plasticity (MBIP) and dynamic slip band refinement (DSBR) [8,9]. The activation of these mechanisms depends on the stacking fault energy (SFE) of austenite, which is controlled by chemical composition, the micro-segregation of alloving elements (Suzuki effect) [10], temperature, and grain size [11-14]. In general, the activation of stress-induced martensitic transformation $(\gamma \rightarrow \varepsilon)$ occurs in steel alloys with SFEs values lower than 20 mJ/m^2 [15,16],

whereas deformation twinning has been observed within the range of $20-40 \text{ mJ/m}^2$ [16]. Partial and/or perfect dislocation gliding has been observed when SFE values are above 40 mJ/m^2 , and a predominant MBIP or DSBR effect has been reported in steels with SFEs values above 60 mJ/m^2 [7,9,17,18].

The existing literature shows that the deformation mechanisms in manganese steels have been mostly studied experimentally through a standard methodology composed of tensile tests in combination with TEM [19–23], XRD [24,25], or in situ neutron diffraction [26]. When this approach is not available, the nanoindentation technique could be used to collect indirect evidence about the deformation mechanisms. During a nanoindentation test, the indenter induces a plastic deformation on the surface material. If the local deformation energy is sufficiently high, a nucleation-controlled transformation can occur that induces a volumetric contraction. When this occurs, the surface separates from the tip and the indenter control system ceases sensing the reaction force, thus showing a change in the load-displacement curve. Once the nanoindenter tip re-establishes contact with the surface, it resumes from the last applied force magnitude but at a higher displacement value, revealing a feature known as a "pop-in".

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SFE

DRX

 σ_{c}

 σ_p

 ε_c Ė

d

σ

 T_d dDRX

hf OIMs

Nomenclature

grain size, um

lature	TMT	thermo-mechanical treatment	
	Z	Zener-Hollomon parameter, s ⁻¹	
stacking fault energy, mJ/m^2	Q	activation energy for hot working, kJ/mol	
dynamic recrystallization	A_1A_2, A_3	n_1, n_2, n_3 material constants	
critical stress, MPa	m	strain rate sensitivity exponent	
peak stress, MPa	α	stress multiplier	
critical strain	η	efficiency parameter	
strain rate, s ⁻¹	R	molar gas constant (8.314 J/(mol K))	

true strain

instability condition

work hardening rate, MPa

grain orientation spread

maximum displacement, nm

DRXed grains dynamically recrystallized grains

ε

ξ

θ

 h_{max}

GOS

This phenomenon allows for studying deformation-induced phase
transformation in a more accessible manner. Furthermore, these
pop-ins and their relation to the load can also reveal additional
important metallurgical issues. For instance, the initial pop-in can be
related to the creation and/or nucleation of dislocations in material
considered to begin as defect-free. Thus, the force at which the first
pop-in occurs can determine the theoretical strength of the crystal [27].
Furthermore, the occurrence of subsequent pop-ins in the load-
displacement curve can be related to stress-induced martensitic
transformation $(\gamma \rightarrow \varepsilon)$ as seen in studies by Ahn et al. [28], He et al.
[29] and Misra et al. [27] for high-nitrogen steel, duplex manganese
steel, and stainless steel, respectively.

flow stress of hot compression, MPa

discontinuous dynamic recrystallization

deformation temperature, °C

orientation image micrographs

residual displacement, nm

A notable combination of hardness, strength and ductility has been observed for Fe-Mn-Al-C steel alloys with high C and Al contents. The high ultimate tensile stress (UTS) and high total elongation (TEL) are attributed to the operation of the microband induced plasticity effect (MBIP) due to the high SFE, which during plastic deformation prevent further dislocation slip and result in high-strain hardening [17]. The high alloy content of C and Al promotes the precipitation of coherent nanoscale (Fe, Mn)₃AlC carbides within the austenite (denoted as ĸcarbide or κ -phase [30]) during the aging treatment [4,21,31-33]. These carbides also play an important role in mechanical properties, particularly with hardness. James [34] conducted the first research on κ-carbides and observed the age-hardening effect with increasing amounts of carbon and aluminium in Fe-Al systems. He also found that the highest hardness level was obtained when the aging treatment was performed in a temperature range of 500-550 °C. In the same year, Kayak [35] also proposed aging temperatures between 500 and 550 °C for Fe-Mn-Al systems to obtain the best mechanical properties. Two decades later, Bozhko (1988) [36] obtained a maximum hardness at 550 °C using Fe-29%Mn-9.3%Al-0.95%C steel independent of the aging time, which was varied between 10 and 250 h. These results were consistent with those obtained by Wu et al. [37] and Springer et al. [32], who achieved maximum hardness at 550 °C after 24 h of aging time. Recently, Lee et al. [33] described a dramatic hardening of Fe-31.4Mn-11.4Al-0.89C steel after 16 h of aging at 550 °C. Moreover, other authors have recommended the use of double aging treatments to increase the volume fraction of κ -carbides within the austenitic matrix, as the austenite phase remains in a supersaturated state even after the initial aging treatment [38].

The major metallurgical and mechanical features obtainable for Fe-Mn-Al-C steel alloys significantly depend on the applied thermomechanical treatments; during the hot working process, dynamic recrystallization (DRX) is the most important phenomenon controlling the resulting microstructural and mechanical properties. Dislocation climb and cross slip are promoted with a high SFE, thus encouraging a dynamic restoration process known as dynamic recovery (DRV). When the SFE is low, dislocation movement is restrained and an inhomogeneous dislocation density distribution is promoted that leads to

discrete nucleation sites for new grains. This last mechanism in known as discontinuous dynamic recrystallization (dDRX) [39]. The onset and propagation of dDRX depend on the chemical composition of the alloy, the grain size prior to deformation, the mode of deformation, and the deformation conditions [39-41]. The initiation of dDRX is typically characterized through the critical stress (σ_c) and strain (ε_c) that are influenced by deformation parameters such as temperature (T), strain (ε), and strain rate ($\dot{\varepsilon}$). These two critical variables can be experimentally determined through metallography, but this requires extensive sampling before and after the critical strain. However, the critical stress and strain can be analytically determined by interpreting the hot flow curves through mathematical models such as those proposed by Poliak and Jonas [42] or Ryan and McQueen [43].

At present, virtually no investigations have examined the hot working of kappa Fe-Mn-Al-C steel. Consequently, this work studies the hot working behaviour of kappa-formed steel and its deformation mechanisms through nanoindentation. In addition, this work reviews the effect of aging and thermomechanical treatments on deformation mechanisms, which has seldom been addressed in the existing literature

2. Materials and methods

2.1. Material chemistry

The steel used in this investigation was produced in an open induction furnace at the Foundry Laboratory of the Metallurgical Research Institute at UMSNH (Morelia, México). The chemical composition was determined using a Bruker model Q4-Tasman arc spark spectrometer and is shown in Table 1.

The SFE of the steel was estimated at $\approx 92 \text{ mJ/m}^2$ at room temperature using a sub-regular solution thermodynamic model for Fe-Mn-Al-Si-C steels optimized by Zambrano [44]. The possible effect of the initial austenitic grain size on the SFE, which is still under examination, was taken into account for this calculation [45,46].

2.2. Aging treatment

A thermomechanical treatment was proposed for this work; to compare the effects of deformation mechanisms and hardness, a convectional aging treatment was performed on the Fe-Mn-Al-C steel after solution treatment at 1150 °C for 10 h in an argon atmosphere

Table 1

Chemical composition of Fe-Mn-Al-C steel with high-carbon and high-aluminium content (in wt%).

Mn	Al	Si	С	Fe
20.71	11.12	1.98	1.55	Bal.

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