

A study of microstructure and phase transformations of CMnAISi TRIP steel

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<u>ABSTRACT</u>

Purpose: Purpose was to obtain the TRIP-type microstructure in the CMnAlSi steel. Heat treatment consisted of the partial austenitization at 900°C/60s and continuous cooling with rates: 0.5-40°C/s, was examined. Also the effect of Al and Si on Ac1 and Ac₃ temperatures, and the volume fractions of austenite in CMnSi, CMnAl and CMnAlSi steels was investigated.

Design/methodology/approach: The effect of alloying elements on Ac_1 and Ac_3 temperatures was investigated using Thermo-calc program. The influence of cooling rates on phase transformations and microstructures of samples austenitized at 900°C/60s was examined using dilatometer, light optical microscopy and scanning electron microscopy. X-ray diffraction technique was used to calculate the amount of retained austenite. Quantitative analyses of phases were done using Image pro Plus 3.0 program. The mechanical properties and Vickers hardness (HV10) measurements were also investigated.

Findings: The TRIP-aided microstructure consisted of ferrite matrix, bainitic ferrite and metastable retained austenite can be obtained for the CMnAlSi steel through intercritical annealing at 900°C/60s and continuous cooling with the rate 20°C/s to the R.T. Isothermal holding at bainitic temperature range (600-400°C) during cooling is not necessary, because of the Al and Si additions to the steel.

Practical implications: The CMn steel with addition of 1% Al and Si is well-suited for production of TRIP steel sheets in a large range of temperatures: 800-900°C. The advisable cooling rates are in the range from 10 to 40°C/s. **Originality/value:** In the TRIP steels the amount of residual austenite in structure at the R.T. strongly depends on the heat treatment parameters such as annealing temperature, cooling rates and amounts of added alloying elements. It is very important to determine the optimal annealing parameters for each TRIP steel grade to obtain the steel with the best mechanical properties and microstructure.

Keywords: Heat treatment; Automotive steel; TRIP microstructure; Intercritical annealing

1. Introduction

Engineered steels provide automotive designers and manufacturers with the unique option to combine lightweighting with the traditional steel advantages of low cost.

The catalog of commercial steel grades includes high-strength steels (HSS) and ultra high-strength (AHSS) steels grades. Conventional high strength steels have the yield strength in the range from 210 to 550 MPa, UHSS are defined as steels with yield strength greater than 550 MPa as it is shown in Figure 1.

The principal differences between conventional HSS and AHSS are due to their microstructures. AHSS are multi-phase steels, which contain aside well workable ferrite different shares of martensite, bainite and/or retained austenite in qualities sufficient to produce unique mechanical properties. Conventional steels, such as interstitial free steels, have lower strength and elongation, because of their mainly ferrite microstructure [1, 9].

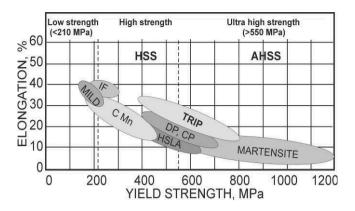


Fig. 1. Strength-formability relationships for mild, conventional HSS and advanced HSS steels [9]

In the last two decades, as one of the advanced high strength steels, TRIP-aided steels have attracted more attention of both the metallurgy industry and the automobile industry. TRIP-aided steels are used especially for automotive applications such as center pillar reinforcements or seat frames.

TRIP steels have a multi-phase microstructure composed of a ferrite matrix, bainite and retained austenite in the amount of about (5-12%).

During tensile elongation, metastable retained austenite transforms into martensite. These tiny martensite islands greatly improve both the strength and the ductility of the steel sheets. This phenomenon is called transformation-induced plasticity (TRIP) [12, 15, 20].

Transformation of retained austenite to martensite can appear by mechanical activation in the elastic or in the plastic region, depending on its thermodynamic stability. As shown in Figure 2 below Ms temperature the γ –> Ms transformation is induced thermally. In the range from Ms to Ms[°] martensite transformation is stress-induced. Strain-induced transformation (TRIP) is observed from Ms[°] to Md. At higher temperature than Md no TRIP effect can be observed. Yield strength of the steel decreases linear with the temperature increasing [7].

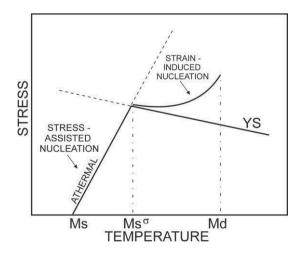


Fig. 2. Thermodynamical stability of retained austenite [7]

The TRIP-aided microstructure can be achieved after intercritical annealing and isothermal treatment [20]. The amount of retained austenite strongly depends on intercritical annealing parameters: temperature, time, cooling rate. During intercritical annealing of the cold-rolled sheet steel the dissolution of cementite, recrystallization of ferrite and ferriteaustenite transformation occurs. Depending on the chemical composition of the steel and austenitization temperature different amounts of austenite are formed. After austenitization, fast cooling in the pearlite transformation region should be applied, in order to avoid the pearlite transformation. In TRIPaided steels with additions of Al or/and Si the precipitation of carbides from the austenite is prevented. Austenite transforms in the bainite temperature range to bainitic ferrite, the rest austenite remains in the microstructure as metastable retained austenite [10, 11, 16]. Increasing the amount of bainitic ferrite results in better stabilization of the retained austenite due to mechanical effect from the surroundings of the austenite. The austenitization temperature has an influence on the mechanical properties of the TRIP steel in the range 730-780°C. The increase of the annealing temperature results in increasing strength and elongation values due to incomplete dissolution of cementite at temperatures below 780°C for the high heating rates. Increasing the temperatures further results in slightly decreased strength level and increased elongation values. This could be explained by the grain size, which generally increases with increasing temperature [2, 3, 5, 8, 14, 17-19].

Also, the chemical composition is very important of the C-Mn steels. Si is the conventional alloying element in TRIP steels. It is known, that high Si levels are not well suited to such industrial practices as galvanizing and hot rolling. Some studies dealing with others elements, such as Cu, Ni, Al, Mo and P can be found in the literature [4, 13]. In the work authors have shown the effect of alloying elements on the phase transformations of the CMn steels.

2. Material and experimental procedure

The chemical composition of investigated TRIP steel is presented in Table 1.

Table 1.						
Chemical composition of the investigated steel						
Steel	С	Mn	Al	Si	Р	N
CMnAlSi	0.150	1.550	1.090	1.010	0.013	0.003

In order to calculate the A_{c1} and A_{c3} temperatures of the investigated CMnAlSi steel for equilibrium conditions, Thermocalc program was used. Data were verified experimentally using dilatometer. Samples were continuously heated with the rate 180°C/s to the temperature 1200°C and continuously cooled with the same rate to the R.T. The results for investigated CMnAlSi steel were set-up with the results for the CMnAl and CMnSi steels from literature [17]. The chemical compositions of CMnAl and CMnSi steels from literature data [17] are shown in Table 2.

Table 2.						
Chemical	compositio	ns of TRI	IP steels f	from liter	ature data	ı [17]
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Steel	С	Mn	Al	Si	Р
CMnSi	0.25	1.67	0.03	1.28	0.012
CMnAl	0.18	1.56	1.73	0.02	0.017

The volume fractions of austenite as a function of temperature of CMnSi, CMnAl [17] and investigated CMnAlSi steel are shown in the Figure 3. In the investigated CMnAlSi steel the Ac₁ temperature is higher of about 25°C than in the CMn steel with addition of 1.28% of Si and equals 724°C.

In the CMn steel with addition of 1.73% Al, it is not possible to obtain fully austenitic region. In the CMnAlSi steel Ac_3 temperature equals 1148°C [15].

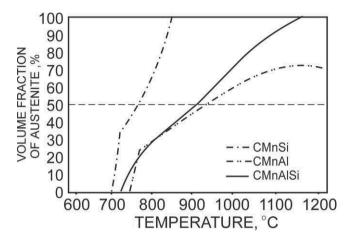


Fig. 3. Volume fraction of austenite as a function of temperature: equilibrium calculations of CMnSi, CMnAl [17] and the investigated CMnAlSi steel

The initial heat treatment of investigated CMnAlSi steel is shown in Figure 4. Steel was hot rolled in temperatures 1150, 1100 and 1000°C and slowly cooled on air to the R.T. The steel sheet was then cold-rolled with 75% section reduction to the thickness of 1.14 mm.

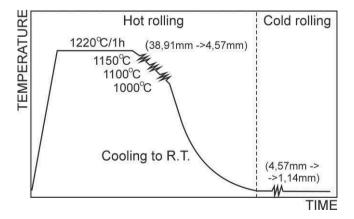


Fig. 4. Schematic diagram of hot and cold rolling procedure of CMnAlSi steel

In the work the influence of cooling rate on phase transformations after intercritical annealing at 900°C was investigated. The dilatometric samples (10 x 5 x 1 mm) from the CMnAlSi steel sheet were processed in 805 DIL dilatometer with inductive heating. Samples were heated with the rate 20°C/s to the temperature 900°C austenitized during 60 s and continuously cooled with the various cooling rates from 40 to 0.5°C/s to the R.T.

The data from dilatometric curves allowed for creation of CCT diagram of TRIP-aided CMnAlSi steel.

Microstructures of the samples were investigated using light optical microscope Neophot 32 and scanning electron microscope SEM-EDX Philips JSM-5400.

Image Pro Plus 3.0 program was used to the quantitative analysis of microstructures observed at magnification 1000x (OM) for the ferrite-pearlite samples in the initial state, and at magnification 1500x (SEM) for heat treated samples, because of the refinement of the grain size of ferrite after intercritical annealing.

The presence of retained austenite in the microstructure was investigated using X-ray diffraction method on Philips diffractomether. The volume fraction of austenite was calculated using reflexes comparison method [6].

YS and TS of the ferrite-pearlite and TRIP samples were examined using ZWICK Z100 tensile test machine with digital controller. Also Vickers hardness HV10 measurements were shown.

3. Discussion of results

Thermo-calc program was used to create the equilibrium diagrams of various CMn steels (0.15 wt. % C) with additions of Al and/or Si in the range from 0 to 1.5 wt. %. Obtained data allowed for creation of diagrams which are shown in Figures 5 and 6. In Figure 5 the influence of the Al addition on A_{c1} and A_{c3} temperatures in CMnSi (0.15% C, 1.55% Mn, 1.01% Si) steel was presented.

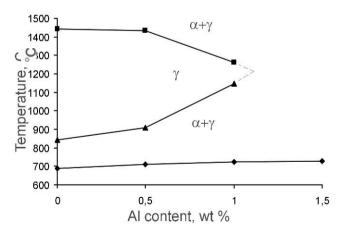


Fig. 5. The influence of the Al content on A_{c1} and A_{c3} temperatures for CMnSi steel (0.15% C, 1.55% Mn, 1.01% Si)

In Figure 6 the influence of the Si content on A_{c1} and A_{c3} temperatures for CMnAl steel (0.15% C, 1.55% Mn, 1.09% Al) is shown. It can be seen that Al and Si haven't got the strong

influence on A_{c1} temperature, it varies from 688 to 733°C. Increasing of Al and/or Si content in the steel causes the considerable growth of the A_{c3} temperature. It is not possible to obtain the fully austenitic region for the steel with the addition of Al or Si higher than about 1.2%. Al and Si are the elements strongly stabilizing ferrite [4, 15]. The A_{c1} and A_{c3} temperatures of investigated CMnAlSi steel equal respectively 724°C and 1148°C [10, 15].

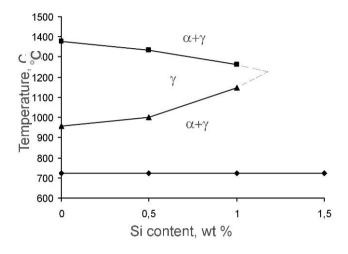


Fig. 6. The influence of the Si content on A_{c1} and A_{c3} temperatures for CMnAl steel (0.15% C, 1.55% Mn, 1.09% Al)

Microstructure of the steel after hot and cold rolling, according to scheme in Figure 4 consisted of ferrite and pearlite. The amount of ferrite was about 78%. Optical micrographs of the sample in the initial state in the rolling direction (L) and in the transverse (T) direction have been shown in Figure 7 and 8 at magnification 500 x.

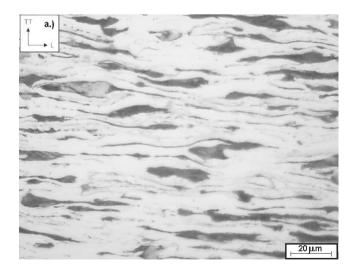


Fig. 7. Optical micrograph of experimental starting cold rolled microstructure in the rolling direction (L)

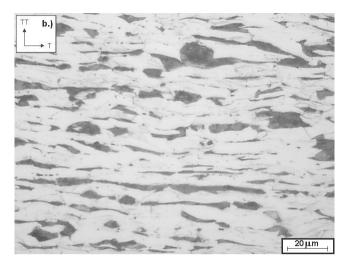


Fig. 8. Optical micrograph of experimental starting cold rolled microstructure in the transverse direction (T)

During continuous annealing at 900°C at the $(\alpha+\gamma)$ temperature range two metallurgical mechanisms took place: recrystallization of the work hardened ferrite and partially austenitization during the holding at intercritical temperature. The recrystallization of C-Mn steels appears in the very early steps during intercritical holding; therefore it will not be discussed in this article. For austenitization, two different nucleation sites are theoretically possible, such as ferrite/ferrite grain boundaries and ferrite/cementite interfaces.

Austenite nucleation on the ferrite/cementite interfaces as shown in Figure 9 is thermodynamically encourages by the high local carbon and manganese source compared to the ferrite/ferrite grain boundaries. In case of cementite at the ferrite junction points, the nucleation rate is higher compared to cementite in the ferrite matrix, what is caused by the additional surface energy available. As it was studied by [12, 16, 18] the austenite nucleus rapidly envelops the cementite particle. The process can be seen in the Figure 10 for the sample austenitized at 750°C/60s and cooled with the rate 20°C/s.

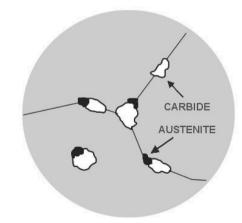


Fig. 9. Austenite nucleation sites at cementite

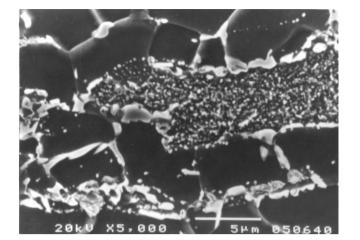


Fig. 10. Partial austenitization of cementite in the microstructure of the sample austenitized at $750^{\circ}C/60s$

During austenitization at 900°C/60s, in the $(\alpha+\gamma)$ temperature range, the amount of about 50% of austenite can be obtained. This carbon and manganese-rich phase transforms during cooling to ferrite, pearlite, bainite or/and martensite depending on applied cooling rate. The dilatometric curves allowed for creation of CCT diagram of investigated steel which is shown in Figure 11.

On the base of CCT diagrams of the CMnAlSi steel for the partial austenitization at temperatures 900°C (Fig. 11) and 800°C [15] can be stated, that in order to obtain typical multiphase TRIP microstructure with polygonal ferrite matrix, bainitic ferrite and retained austenite (γ_R) (Fig. 12), cooling rates in the range from 10 to 20°C/s should be applied.

Additions of about 1 wt.% of Al and 1 wt.% Si prevent the precipitation of carbides in bainite, enriching the austenite in carbon, which allows remaining of about 10% of metastable retained austenite in microstructure at the R.T. For the higher cooling rates the martensite transformation occurs, which reduces the volume fraction of retained austenite in the microstructure. Also the pearlite occurrence is unadvisable, it's transformation is observed after cooling with rates slower than 10°C/s.

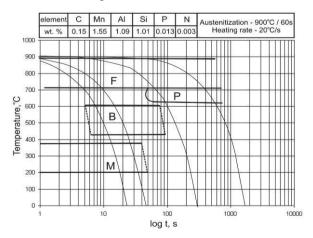


Fig. 11. CCT diagram of investigated CMnAlSi steel after austenitization at 900°C/60s, at (α + γ) temperature range

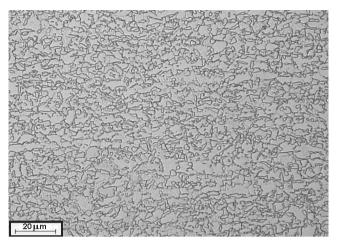


Fig. 12. TRIP type microstructure of the sample cooled from 900° C/s with the rate 20° C/s, OM, 500x

The precise analyses of constituents were done with scanning electron microscopy (SEM). The micrographs of the samples after particular heat treatments are shown in Figures 13-16.

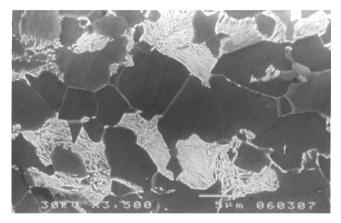


Fig. 13. SEM micrograph of the sample cooled from 900°C/60s with the rate 0.5°C/s

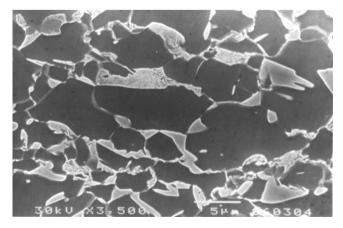


Fig. 14. SEM micrograph of the sample cooled from 900°C/60s with the rate $3^{\circ}C/s$

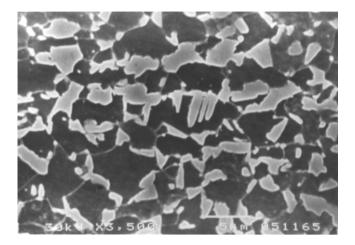


Fig. 15. Typical TRIP microstructure of CMnAlSi austenitized at 900°C/60s cooled with the rate 20°C/s, SEM

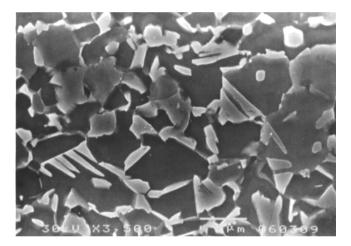


Fig. 16. SEM micrograph of the sample cooled from 900°C/60s with the rate 40° C/s

The X-ray diffraction analysis revealed the occurrence of ferrite (0.202 nm, 0.143 nm, 0.117 nm) and austenite (0.207 nm) reflexes of the samples heat treated at 900°C/60s. In the ferrite-pearlite samples only ferrite was found.

The Toray equation allowed for evaluation of the amount of retained austenite (γ_R) in samples. The amount of about 10% of γ_R in the sample cooled with the rate 20°C/s was found (Fig. 17). Samples cooled with higher or lower cooling rates had lower amount of retained austenite (about 2-7%).

Image Pro Plus program allowed for evaluation of the volume fractions of various phases in the microstructure of the CMnAlSi steel. In cold-rolled sample the amount of 22% of pearlite was found in the ferrite matrix. Microstructures of the heat treated samples consisted of about 79% of ferrite, and 21% of other phases. In the sample cooled with the rate 0.5° C/s the whole austenite transforms into ferrite and pearlite. After cooling with the rate 20° C/s austenite transforms into ferrite, bainitic-ferrite (11%), the rest of austenite remained as retained austenite (10%). For the highest cooling rate (>40^{\circ}C/s) austenite transforms mainly to martensite.

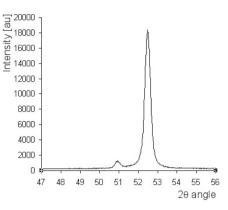


Fig. 17. X-ray diffraction of the sample cooled from $900^{\circ}C/60s$ with the rate $20^{\circ}C/s$

After intercritical annealing at 900°C/60s the high refinement of the mean chord of ferrite is observed in comparison with the mean chord of ferrite in the initial state (15 μ m). The increase of cooling rate from 0.5 to 40°C/s, caused the decrease of the mean chord of ferrite grains from 3.4 to 2.3 μ m. The mean chords of new phases (P, BF, M) varied from of about 1.5 to 1.8 μ m [10]. In Table 3 the ferrite grain size parameters such as mean chord and mean area, are listed. Distributions of measured ferrite mean chords for various heat treated samples are shown in Figure 18.

Table 3.

Mean chords of ferrite of the heat treated samples

Sample	Mean chord of	Mean area of ferrite	
	ferrite [µm]	$[\mu m^2]$	
0.5°C/s	3.4	12.7	
3°C/s	2.9	9.7	
20°C/s	2.8	9.2	
40°C/s	2.3	5.3	

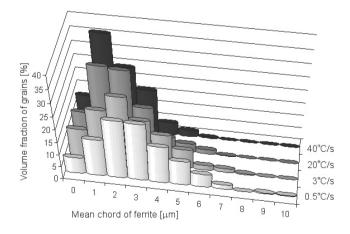


Fig. 18. Mean chord of ferrite histograms of samples cooled from $900^{\circ}C/60s$ with rates 40, 20, 3 and $0.5^{\circ}C/s$

The nominal stress – strain curves of the cold-rolled ferritepearlite sample and intercritically annealed TRIP sample are shown in Figure 19. Mechanical properties such as YS and TS of samples are listed in Table 4. Typical ductile fracture of the CMnAlSi steel is shown in Figure 20.

The Vickers hardness HV10 of the cold rolled steel equaled 300 HV10. Results of hardness measurements of the samples cooled with various cooling rates from 900°C to R.T. are shown in Figure 21.

Table 4.

Mechanical properties of the cold rolled initial sample and heat treated at 900°C/60s TRIP sample

Sample	YS _{0.2} [MPa]	TS [MPa]	$A_g[\%]$	A [%]
Cold rolled	1002.8	1066.5	1.1	3.7
TRIP	304	806.5	15.9	21.1

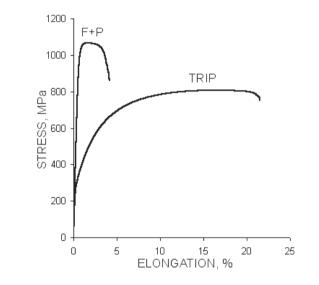


Fig. 19. Nominal stress – strain curves of CMnAlSi steel a) in the initial state after cold rolling (ferrite-pearlite structure), b) after heat treatment at 900°C (TRIP-aided structure)

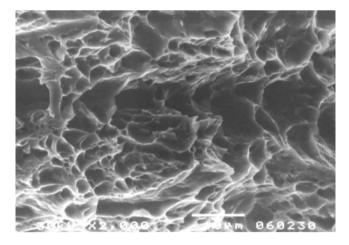


Fig. 20. Scanning electron micrograph of fracture surface after static tensile test of the sample in the initial state

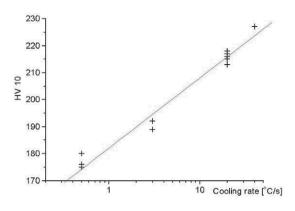


Fig. 21. Vickers hardness measurements of the samples cooled from $900^{\circ}C/60s$ with various cooling rates

4.Conclusions

The CMnAlSi steel in the initial state had the ferrite-pearlite cold-rolled band microstructure with 22% of pearlite.

During heating and annealing at 900°C the recrystallization of work hardened ferrite, dissolvation of cementite and partial austenitization take place.

After 60 s of austenitization at 900° C duplex-type microstructure consisted of 50% of austenite and ferrite is obtained.

TRIP-type microstructure consisted of ferrite matrix (79%) with the bainitic ferrite (11%) and metastable residual austenite (10%) can be obtained after cooling with the rate 20° C/s.

Additions of 1 wt. % of Al and Si prevent the precipitation of carbides in bainite, so the bainitic ferrite is observed in the microstructure of samples cooled with the rates 20-40°C/s.

After cooling with low cooling rates (0.5-3°C) the ferritepearlite microstructure is obtained.

For the higher cooling rates (>20°C/s) the martensite is observed in the microstructure.

The steel, after austenitization at 900°C/60s at the $(\alpha+\gamma)$ temperature range and continuous cooling to the R.T. has the ultra fine-grained microstructure. The mean chord of ferrite for the TRIP type samples (40-20°C/s) equaled respectively 2.3-2.8 μ m. Mean chords of retained austenite, bainitic ferrite and martensite equaled from 1.5 to 1.8 μ m.

Vickers hardness of the cold-rolled sample in the initial state equaled 300 HV10. The TRIP type microstructures had the hardness from 215 to 228 HV10.

YS and TS of the ferrite-pearlite cold-rolled sample equaled respectively 1003 and 1067 MPa. TRIP-type sample annealed at 900°C had the YS and TS respectively 304 and 807 MPa. The total elongation of the TRIP-type sample increased from 3.7 to 21% in comparison with the cold rolled sample.

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