



INNOVATIVE HOT DIP GALVANIZING PROCESS USING ZINQUENCH FOR PROCESSING ADVANCED HIGH STRENGTH STEELS¹

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Abstract

ZinQuench - Dynamic Galvanizing[®] (ZQ-DG) is an approach combining coatability improvement and process flexibility to produce advanced high strength steels (AHSS). During this process, steel strips are gas-jet cooled and quenched from a high tuned temperature into a zinc melt with dynamic flow after annealing. The scientific objective is to develop ZQ-DG to design new AHSS with improved coating quality and mechanical properties simultaneously. The influence and effectiveness of the theoretical considerations are tested experimentally. Process parameters are studied with a laboratory scale hot-dip process simulator. Mechanical characterization of processed multiphase AHSS is carried out through tensile test and correlated with steel microstructure. Wettability is also evaluated. Actual state of the investigations and results are presented and discussed for different steels processed. Comparison between laboratory ZQ and conventional hot dip galvanizing results allows forecasting industrial application potential of ZQ-DG to process flexibly new galvanized AHSS with lower alloying element content and resulting in less expensive products with improved mechanical properties.

Keywords: Hot-dip galvanizing; Advanced high strength steels; ZinQuench.

PROCESSO INOVATIVO DE ZINCAGEM POR IMERSÃO A QUENTE USANDO ZINQUENCH PARA O PROCESSAMENTO AVANÇADO DE AÇOS DE ALTA RESISTÊNCIA

Resumo

ZinQuench – Dynamic Galvanizing[®] (ZQ-DG) é um metódo para zincagem de aços de alta resistência (AHSS) que combina a ótima espalhabilidade do recobrimento e a flexibilidade do processo. Durante este processo chapas de aço são resfriadas a gás depois do aquecimento e imergidas em zinco fundido através de um fluxo dinâmico. O objetivo científico deste trabalho é o desenvolvimento de ZQ-DG para novos designs de AHSS com um recobrimento de ótima qualidade e com melhores propriedades mecânicas. A influência assim como a eficiência das considerações teóricas são testadas experimentalmente. Os parâmetros do processo são estudados com um simulador de laboratório para zincagem por imersão a quente. Análises mecânicas dos aços processados são realizadas através de ensaios de tração e correlacionadas de acordo com a microstrutrura. A molhabilidade é também caracterizada. O estado atual das investigações e dos resultados são apresentados e discutidos para differentes acos processados. Uma comparação entre os resultados do ZQ de laboratório e o método convencional de zincagem por imersão a quente permitirá uma previsão da aplicação industrial do ZQ-DG para novos AHSS, processados flexivelmente com baixo conteúdo de elemento de liga e resultando em produtos mais baratos com melhores qualidades mecânicas.

Palavras-chave: Zincagem por imersão a quente; Aços de alta resistência; ZinQuench.

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1 INTRODUCTION

Recent years have seen many new developments in steel technology and manufacturing processes to build vehicles of increased safety and crash performance, while reducing mass and cost, with Advanced High-Strength Steel (AHSS) grades. These AHSS are multiphase steels, like dual phase (DP) and transformation-induced plasticity (TRIP) steels. DP steels consist of a ferritic matrix containing a hard martensitic second phase in the form of islands. The microstructure of TRIP steels is retained austenite (min. of 5 vol.%) embedded in a primary matrix of ferrite-bainite.

During the last twenty years, continuous hot dip galvanizing (HDG) became widely accepted as an all-important strip production process, since it combines at the same time recrystallisation annealing and Zn coating in one unique process. At present most multiphase steels use high amounts of alloying elements to ensure specialized microstructure and properties making these grades very costly and difficult to process. These multiphase AHSS grades make hot dip galvanizing very difficult due to the poor and inhomogeneous wettability resulting in bare spot defects. The AHSS poor coatability results from segregations of the various alloying elements (like Mn and Si) at the steel surface occurring during recrystallisation annealing. The effect of the annealing atmosphere on the surface metal oxides resulting from alloying elements segregations can be controlled through the atmosphere properties to some extent.^[1-3]

A new approach for coatability improvement of AHSS would be to use the ZinQuench - Dynamic Galvanizing[®] (ZQ-DG) technology, which is a continuous hotdip galvanizing process. During this innovative process, steel strips are quenched in the liquid zinc directly after recrystallisation annealing, the zinc melt being continuously circulating and promoting tubular flow.^[2-8]

The aim of the scientific work is to investigate ZQ-DG process for the design of new AHSS, like DP and TRIP steel grades, with superior coating guality and improved coated product properties. Namely, the objectives are to optimize ZQ-DG process parameters to reach improved steel mechanical properties and also improved Zn laver at the same time, in comparison to conventional HDG process. These theoretical considerations are tested experimentally through laboratory scale investigations to evaluate the projected improvements.

Actual state of the investigations and results are presented and discussed in this publication, the focus being put on mechanical properties of different AHSS paper. Reachable mechanical properties, microstructures and coating properties during laboratory ZQ and conventional HDG are analysed and compared to establish critical process influence factors that yield outstanding AHSS grades with improved coated surface.

2 MATERIALS AND METHODS

2.1 ZinQuench - Dynamic Galvanizing (ZQ-DG)

ZinQuench - Dynamic Galvanizing[®] (ZQ-DG) technology is a continuous hot-dip galvanizing process. Steel strips are guenched in the liquid zinc with ΔT_{max} = 100°C (ΔT : T_{strip} – T_{Zn bath}), by this a cooling rate of dT/dt _{max} >> 100 K/s can be realized. The zinc melt is continuously cooled and circulating, promoting tubular flow.

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Fundaments and highlights of the ZQ-DG concept are that: 1- With this process the steel tensile strength can be raised even though using less alloying elements, since higher cooling rates for microstructure setting are achieved during quenching in the Zn bath compared to conventional HDG. 2- Simultaneously the coatability, i.e. surface reactivity and wettability, with Zn can be improved, since high strip immersion temperature is processed and enhances the activation energy for aluminothermic reaction. 3- Also superior surface Zn coating morphology and quality can be processed, since the Zn pot design with continuous dynamic melt flow avoids located depletion of bath AI and thus allows improved intermetallic Fe-AI inhibition layer formation at the substrate/coating interface.^[4-8]

2.2 Materials

The chemical compositions in mass contents and sheet thicknesses of the tested materials are summarized in Table 1. All steel grades were industrially cold rolled sheets supplied in full hard condition. The TRIP steels differ in their alloying concept.

	Chemical compositions, mass content in %									
Steel grade	С	Si	Mn	Cr	Мо	AI	Р	Nb	Ti	V
DP (1.2 mm)	0.07	0.12	1.40	0.730	<0.010	0.05	<0.01	-	-	-
Si-TRIP (1.3 mm)	0.19	1.61	1.47	0.023	<0.005	0.05	0.01	<0.001	0.007	0.007
AI-TRIP (1.0 mm)	0.22	0.10	1.59	0.017	<0.005	1.46	0.01	<0.001	0.005	0.006
Si-Al-TRIP (1.5 mm)	0.20	0.62	1.45	0.012	<0.005	1.06	0.01	<0.001	0.005	0.007

Table 1. Chemical compositions (mass contents in %) and sheet thicknesses of the investigated steel grades. (Nb, Ti and V not determined for DP)

2.3 Hot Dip Process Simulator

The laboratory study of hot dip galvanizing is carried out using a modified hot dip process simulator at RWTH Aachen University (Rhesca Hot Dip Process Simulator EU A II from Iwatani Corp.). It simulates the annealing and hot dipping process of steel strip in accordance with the commercial production of hot dip coated steel strip and enables the development of new steel grades. Steel sheet samples are heat treated and subsequently coated by hot dipping in laboratory scale. An overview of the total configuration is shown in Figure 1.

Sample temperature is controlled with the help of a thermocouple spot-welded on the sample surface. Process gas is supplied by a gas mixing station. The atmosphere of the furnace section during annealing can be set under programme control. The dewpoint of the atmosphere can be set in the range between -60°C to +10°C. The melting bath is separated by a pneumatically operated gate valve. The lower chamber is permanently purged using N_2 as protective gas during operating. Wiping nozzles are mounted to control the thickness of the coating using N_2 as wiping gas.

The geometry of the steel sheet samples (120 x 200 mm²) is given in Figure 2. The so-called homogeneous area is where the temperature distribution is optimised using two additional thermocouples as indicated (mark T1 and T2).





The present experiments are carried out without bath agitation, the top dross on the surface of the bath is removed using the dross removing paddle. The zinc bath design of the hot dip process simulator will be modified in order to ensure dynamic galvanizing (DG) during ZQ. The melting bath will be equipped with a bath agitation system: a zinc stirring equipment will be mounted in the crucible, allowing a Zn movement in a duct, Figure 3.



Figure 1. Layout of the Hot Dip Process Simulator at RWTH Aachen University.



Figure 2. Sample sheet geometry with indicated homogeneous area.



Figure 3. Schematic view of bath stirring equipment concept.







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All hot-dip galvanizing cycles are processed in the hot-dip process simulator without bath agitation using a 95% $N_2 - 5$ % N_2 annealing atmosphere with a dew point of +10°C to produce laboratory coated samples. The Zn bath contains 0.20 %Al and samples are dipped during 3 s. Such high dew point favours reactive wetting, since lower dew points like -30°C experimentally showed poor reactive wetting resulting from the selective oxidation of alloving elements at the steel surface occurring during recrystallisation annealing.

2.4 Conventional Hot-dip Galvanizing (HDG) Cycles

The conventional hot-dip galvanizing process parameters, with intercriticalannealing conditions achieving recrystallized microstructure, are summarized in Figure 4 for the different materials. Zn bath temperature was kept at 460°C.

Intercritical annealing temperatures are situated over A1-temperature in the austenite-ferrite two-phase region. Due to their higher alloying, cooling rates for TRIP steels are set lower than for DP.



Figure 4. Conventional hot-dip galvanizing process parameters.

2.5 Influence of ZinQuench Parameters

Heat treatments with regards to the identified intercritical-annealing conditions followed by isothermal holding steps in the temperature region of T>500°C are accomplished varying holding temperature in order to identify the influence of ZQ parameters on mechanical properties and phase transformation.

Processed ZQ cycles and parameter variations are summarized in Figure 5 for the different materials: investigated ΔT (T_{strip} – T_{Zn bath}) are 60°C, 80°C and 100°C. Zn bath temperature was kept at 450°C. Concerning the TRIP steel grades, two different bainitic steps (BS) are tried out: 350°C and 400°C.



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Figure 5. Processed ZQ cycles and parameter variations.

2.6 Analysis

2.6.1 Mechanical properties

Characterization of mechanical properties is realized by uniaxial tensile test according to DIN EN 10002. Tensile test specimens are A50-samples, A80 elongation at fracture is calculated according to EN ISO 2566-1.

2.6.2 Metallography

Microstructures are characterized by metallographic analysis using optical light microscopy. Samples are cross-section embedded, polished and etched with Nital, and additionally with $Na_2S_2O_5$ (bright gray ferrite, dark martensite and bainite, white austenite) for TRIP steels, before being observed with 1000x magnification. Ferrite grain size is determined according to ISO 643. Phase analysis is realized via interactive and quantitative image analysis.

2.6.3 Wettability and coatability

Coatings of the produced samples are analyzed using optical macro inspection. Sample pictures are taken to evaluate coating quality and uniformity (presence of bare spots...) and unwetted bare spots analysis is carried out in the sample homogeneous area by numerical scanning and using ImageJ software to determine the area fraction and average size of bare spots by grey scale value.

3 RESULTS

3.1 Mechanical Properties

The mechanical properties of the different coated steels processed, like $R_{p0,2}$, R_m , A_g , A_{50} and A_{80} -calculated, are summarized in Table 2.

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Table 2. Mechanical properties of coated steels processed

Steel grade	Galvanizing process	R _{p0,2} [MPa]	R _m [MPa]	А _g [%]	A ₅₀ [%]	A ₈₀ [%]	Classification
DP	Conventional HDG	263	542	20,0	27,1	24,4	DP500
	ZQ from 510°C	285	591	18,3	25,5	22,9	DP500
	ZQ from 530°C	266	610	17,7	24,8	22,3	DP600
	ZQ from 550°C	257	589	17,4	24,7	22,2	DP500
Si-TRIP	Conventional HDG	477	621	17,8	25,6	23	-
	ZQ from 510°C , BS at 350°C	496	618	19,4	28,1	25,2	-
	ZQ from 530°C , BS at 350°C	411	632	-	-	-	-
	ZQ from 550°C , BS at 350°C	437	653	-	-	-	-
AI-TRIP	Conventional HDG	513	764	22,5	27,1	24,3	TRIP690
	ZQ from 510°C , BS at 350°C	468	811	22,8	28,8	25,9	TRIP780
	ZQ from 530°C , BS at 350°C	455	829	20,7	23,8	21,3	TRIP780
	ZQ from 550°C , BS at 350°C	436	779	-	-	-	-
Si-Al-TRIP	Conventional HDG	421	721	24,8	31,3	28,1	TRIP690
	ZQ from 510°C , BS at 350°C	370	759	20,2	25,2	22,7	TRIP690
	ZQ from 530°C , BS at 350°C	369	730	-	-	-	-
	ZQ from 550°C , BS at 350°C	384	789	22,7	26,9	24,2	TRIP780

Tensile samples breaking prematurely before uniform elongation can not be analyzed and thus no statistical analysis of elongations is carried out in Table 2 for some processed cycles, since too few evaluable samples are available. Moreover, Si-TRIP samples have too low fracture elongation to be classified as TRIP590.

Mechanical properties comparison between conventional HDG and ZQ processes is presented in Figure 6 for the different materials excluding Si-TRIP (presented TRIP steel grades are ZQ samples processed with a bainitic step BS at 350°C). The strip immersion temperature when dipping T_{strip} is the main influencing factor when comparing both processes: $T_{strip} = 460^{\circ}$ C during conventional HDG and $T_{strip} > 500^{\circ}$ C during ZQ.







Figure 6. Influence of strip immersion temperature on the mechanical properties.

3.2 Microstructure

The micrographs of the processed DP steel are all very similar, independently from the galvanizing process. The observed microstructure is a ferrite matrix with mainly martensite islands (around 25%); ferrite grain size is $6 \mu m$.

Processed TRIP steels have a ferrite-bainite matrix with retained austenite islands; Si-TRIP and Si-AI-TRIP have a grain size of 5 μ m, AI-TRIP of 3 μ m. The retained austenite content could only be estimated because of the very fine islands, estimations are given in Table 3.

Steel grade	Galvanizing process	Retained austenite content
Si-TRIP	Conventional HDG	< 5 %
	ZQ	< 5 %
AI-TRIP	Conventional HDG	~ 5 %
	ZQ from 510°C , BS at 350°C	~ 5 %
	ZQ from 530°C and 550°C , BS at 350°C	> 5 %
Si-Al-TRIP	Conventional HDG	~ 5 %
	ZQ from 510°C and 530°C , BS at 350°C	~ 5 %
	ZQ from 550°C , BS at 350°C	5-10 %

Table 3. Retained austenite content estimations







3.3 Wettability and Coatability

For the DP steel coatability is increasing with ZQ. Although the area fractions of bare spots are very similar when comparing conventional HDG and ZQ, the average bare spot size decreases with increasing T_{strip}. Significant coatability improvement is observed with ZQ from 550°C, Figure 7a.

Concerning Si-TRIP steel, the coatability is also increasing with ZQ, although the coatability with ZQ from 530°C and 550°C are quite similar. ZQ decreases the average size and simultaneously the area fraction of bare spots. Significant coatability improvement is observed with ZQ from 550°C (BS at 350°C), Figure 7b.

Concerning AI-TRIP steel, the coatability is rather slightly increased with ZQ, Figure 7c. But the area fractions and average size of bare spots are very similar when comparing conventional HDG and ZQ samples, independently from T_{strin}. In Figure 7c, the patterning of sample side B after conventional HDG is only a muster originating from sample cooling and should not be considered as an unwetted area.

Concerning Si-AI-TRIP steel, the coatability is increased with ZQ. ZQ decreases the average size and simultaneously the area fraction of bare spots. But the area fractions and average size of bare spots are guite similar and independent from T_{strip} when comparing ZQ samples. Significant coatability improvement is observed with ZQ from 530°C (BS at 350°C), Figure 7d.



DP steel process	Conventi	onal HDG	ZQ from 550°C		
Sample side	А	В	Α	В	
Coated sample picture					
Coating surface quality	+	++	++	++	
Area fraction of bare spots	1.3 %	1.0 %	0.9 %	1.2 %	
Average bare spot size	9.9 mm²	5.3 mm²	3.6 mm ²	4.3 mm ²	

Si-TRIP steel process	Conventi	onal HDG	ZQ from 550°C		
Sample side	Α	В	А	В	
Coated sample picture				L	
Coating surface quality	-		++	++	
Area fraction of bare spots	3.0 %	4.8 %	0.8 %	0.4 %	
Average bare spot size	20.4 mm²	21.9 mm ²	3.6 mm²	3.3 mm ²	

AI-TRIP steel process	Conventi	onal HDG	ZQ from 550°C		
Sample side	Α	В	Α	В	
Coated sample picture					
Coating surface quality	-	++	++	++	
Area fraction of bare spots	2.0 %	0.6 %	0.7 %	0.6 %	
Average bare spot size	3.3 mm²	4.7 mm ²	3.6 mm ²	3.3 mm ²	

Si-AI-TRIP steel process	Conventi	onal HDG	ZQ from 530°C		
Sample side	A	В	А	В	
Coated sample picture	1	A Star			
Coating surface quality	+		++	++	
Area fraction of bare spots	1.2 %	4.5 %	0.6 %	0.6 %	
Average bare spot size	9.2 mm ²	30.7 mm ²	3.9 mm²	4.7 mm ²	

--- very bad, -- bad, - insufficient, + sufficient, ++ good, +++ very good

Figure 7. Coatability comparison between conventional HDG and ZQ processes.



4 DISCUSSION

4.1 Mechanical Properties and Microstructures

If the presented $R_{p0,2}$ values seem to be low for the different materials, this is due to the fact that the materials were not skin pass rolled after coating.

When comparing the results concerning DP steel, tensile strength is around 70 MPa higher when processing ZQ from 530°C instead of conventional HDG. Here DP600 properties are reached through ZQ in comparison to a conventional HDG DP500.

Concerning Si-TRIP steel, additional trials have to be carried out. When processing conventional HDG after critical annealing at 760°C or 780°C, the elongations values obtained are too low ($A_{80} < 23\%$), the tensile strength reaching 621 MPa for 760°C and 778 MPa for 780°C. No significant properties improvements are achieved when processing ZQ after intercritical annealing at 760°C. This is in concordance with the steel microstructure: retained austenite content in processed Si-TRIP samples is too low; TRIP steel should contain at least 5% retained austenite. Additional annealing parameters (intercritical annealing at 770°C for example) will be processed during further experiments in order to achieve satisfying mechanical properties with Si-TRIP.

When comparing the results concerning AI-TRIP steel, tensile strength is around 45 MPa higher with simultaneous improved elongation when processing ZQ from 510°C instead of conventional HDG, even around 65 MPa higher when processing ZQ from 530°C. Here TRIP800 properties are reached through ZQ (even with improved elongation) in comparison to a conventional HDG TRIP700.

When comparing the results concerning Si-AI-TRIP steel, tensile strength is around 65 MPa higher when processing ZQ from 550°C instead of conventional HDG. Here TRIP800 properties with improved elongation are reached through ZQ in comparison to a conventional HDG TRIP700 with improved elongation.

The TRIP steels samples processed with the bainitic step at 350°C show higher tensile strength that these processed at 400°C. This is in agreement with the metallographic investigations: samples with lower bainitic step temperature show a finer and more homogenous microstructure than those processed with higher bainitic step temperature. Also, retained austenite grains seem to be smaller and with a refined repartition.

Concerning the retained austenite content determination, other techniques are substantially more appropriated and precise than optical microscopic analysis. Indeed, Electron Backscatter Diffraction (EBSD) enables a 2D determination delivering information about retained austenite morphology and repartition and X-ray Diffraction (XRD) enables a volume based determination and avoids mistakes related to any sample preparation. Moreover, not only the retained austenite amount is important, but also the retained austenite carbon content and retained austenite stability are of great interest in AHSS. Selected TRIP steel samples will subsequently be analyzed using EBSD in order to study the influence of ZQ parameters on mentioned retained austenite properties.

To explain the ZQ phenomena resulting in tensile strength rising and in yield strength lowering, further microstructure investigations are needed using scanning electron microscope (SEM). Especially for DP steel, microstructure differences with regard to bainite should appear between conventional HDG samples and ZQ





samples when using SEM instead of optical light microscopy, since both coating processes differ in the bainite temperature region.

4.2 Wettability and Coatability

It is obvious that DP steel's wettability is improving with ZQ, Figure 7.

This is related to the aluminothermic reaction induced by the Al present in the Zn bath: $3MnO_{(s)} + 2AI_{(bath)} \rightarrow 3Mn_{(bath)} + Al_2O_{3(s)}$. A higher strip entry temperature is enhancing the activation energy for aluminothermic reactions using Al as a reducing agent for strip surface oxides (Mn-, Si-, Cr-). Aluminothermic reactions are exothermic and thus high activation energy is needed to start them.^[6-7,9-11]

Wettability of Si-TRIP and Si-AI-TRIP steels is also improved when using ZQ, but AI-TRIP steel's wettability improvement when using ZQ is not as remarkable as with Si-TRIP and Si-AI-TRIP.

These phenomena are related to the TRIP steels chemistries and the nature of the related oxides developing during the annealing, which react differently with bath AI during dipping. Depending on the atmosphere dewpoint, present oxides can be MnO, SiO₂ and Mn₂SiO₄ for Si-TRIP steel, MnO, Al₂O₃, Mn₂SiO₄ and SiO₂ or MnAl₂O₄ (depending on Si/AI alloying ratio) for Si-AI-TRIP steel and MnO, Al₂O₃ and MnAl₂O₄ for AI-TRIP steel.^[11-13] By analysing the oxides nature and morphology before and after dipping, it is possible to understand their aluminothermic behaviour and thus the wettability of the mentioned DP and TRIP steels. This will be done in a further investigation stage, also on samples processed with dynamic bath agitation.

Namely, there exist some restrictions for successful aluminothermic reactions when ZQ is processed without dynamic galvanizing (DG): no removal of the reaction products by Zn flow from strip surface would stop the reaction,^[14] significant local consumption of Al would result in located depletion of dissolved bath Al at the bath/substrate interface, promoting inhibition breakdown,^[15] only thin MnO-layer would be reduced by aluminothermic reaction from the strip surface.^[11] With bath agitation, ZQ wettability improvement could be further enhanced^[14,16] and ZQ-DG would also improve the Fe-Al inhibition layer properties present at the steel/coating interface.^[6-8] Thus, the wettability of the different steels has to be investigated with DG bath agitation in order to fully understand their aluminothermic behaviour during ZQ-DG.

Further coating characterizations are also needed in order to evaluate coating properties resulting from ZQ-DG and to compare them with conventional HDG; that will be done on some samples processed with bath agitation. Light optical microscopic inspections will be carried out in order to measure coating thickness (about $10\mu m$), electron microscopy will help to determine Fe-Al inhibition layer properties (uniformity, thickness, morphology, chemical composition) and coating morphology and finally T-bend and ball-impact tests will evaluate coating adhesion.

5 CONCLUSION

When comparing conventional HDG and ZQ processes, higher tensile strengths are achievable using ZQ for a given steel chemistry: tensile strength around 70 MPa higher for DP and TRIP steels. ZQ coated DP600 can be processed from conventional DP500 and TRIP800 from TRIP700. TRIP steels with improved elongations can also be processed with ZQ. Thus, ZQ could be very interesting for processing coated improved AHSS, like from less expensive DP steels without







molybdenum or lean Mn-Si-alloved AHSS, and even for processing Zn coated higher Mn-alloyed ultra high-strength steel grades like martensitic steels.^[2-3,6-7]

Coatability of ZQ coated samples is clearly improved in comparison to conventional HDG samples and the wettability increasing is mostly depending on steel's chemistry. This wettability improvement is related to the aluminothermic reaction induced by the AI present in the Zn bath and enhanced by high strip immersion temperature.

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