



# Influence of nitrogen-doped shielding gas for welding of medium manganese austenites for cryogenic applications

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Received: 25 September 2023 / Accepted: 4 December 2023 / Published online: 12 December 2023  
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## Abstract

The relevance of technical gases is constantly increasing due to environmental and climate policy requirements. The storage and transport of liquefied natural gas (LNG) takes place under cryogenic conditions with substantial volume reduction due to significant storage efficiency. Consequently, there are specific requirements for the mechanical properties of the applied materials at cryogenic temperatures. The materials used so far have been cold-hard, high-nickel austenites, and martensitic steels of the X8Ni9 type. The austenitic materials offer good processing properties, but due to their comparatively low strength and high costs, these materials are not attractive. In contrast, the welding of the martensite has a significant negative impact on the processing quality and the automation of the process. In order to address the problems of automation, material costs, and mechanical properties, particularly high strength and cold toughness, the application of innovative austenitic steels with a medium as well as a high manganese content is suggested. For the qualification of medium manganese austenites as a substitute for maritime applications, the welding processing of an X2CrMnNi17-7-5 (1.4371) is considered under the aspects of the standards and guidelines of the International Association of Classification Societies (IACS), the International Maritime Organization (IMO) and the classification society Det Norske Veritas (DNV). Preliminary investigations have revealed that the application of a conventional filler metal of type G 20 16 3 Mn N L in combination with nitrogen doping of the M12-ArC-2.5 shielding gas according to DIN EN ISO 14175 results in a significant enhancement of the mechanical properties. The addition of 4% nitrogen into the shielding gas caused a diffusion-induced increase in the nitrogen content of the weld metal and a simultaneous increase in strength. Additionally, there were no negative influences on the austenitic microstructure. In consideration of the qualification specifications for welded joints and filler metals of the standards and guidelines according to IACS, IMO, and DNV, the joint welds are examined, and the qualification of the welding process is envisaged. For this purpose, comparative welds of the mentioned material combination are carried out and compared with both the initial gas mixtures and the nitrogen-doped shielding gas.

**Keywords** Nitrogen doping · Medium manganese austenites · Impact strength · Hardness · Microstructure

## 1 Introduction

The demand for industrial gases shows a continuous growth [1]. In particular, liquefied natural gas (LNG) and hydrogen represent an important energy policy instrument for reducing emissions and meeting climate protection targets [2]. The storage and transportation of such materials are primarily in the liquid state under cryogenic conditions and are accompanied by significant volume advantages. Liquefied natural gas has a volume reduced by a factor of 600. Nevertheless, the existing cryogenic conditions are associated with high material and welding processing requirements [3–5]. This applies not only to the strength and notched impact strength required by the classification societies for maritime

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Recommended for publication by Commission II—Arc Welding and Filler Metals.

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application scenarios, but also to the manufacturers' specifications with regard to costs, effort, and automation options.

Up to now, the most common materials have been low-temperature, high-nickel austenitic steels and martensitic steels of type X8Ni9. The austenitic materials have favorable processing properties but are not attractive due to their comparatively low strength and high cost. The welding of martensites, on the other hand, has significant drawbacks in terms of processing quality and the ability to automate the process.

As an alternative, the substitution of the current materials with manganese austenites is being investigated. Due to the application of manganese as an austenite initiator, the proportion of the significantly more cost-intensive nickel can be reduced. The lattice structure allows a high cold toughness of the base material, and the strengths can exceed 700 MPa for high manganese austenites [6–8]. The automation of welding processing can be significantly improved due to the absence of magnetization.

For these applications, the initial focus is on commercially available medium manganese austenites. These are a suitable base material for cryogenic requirements. Welding is carried out with standard shielding gases of type M12 and M14 and conventional filler materials. In the course of the investigations, the doping of small amounts of nitrogen to the shielding gases was considered. Essentially, doping of the shielding gas is a commonly recognized procedure for adjusting the arc and the weld joint [9–11].

The objective is the improvement of the mechanical properties of the weld metal by diffusing nitrogen from the shielding gas. Nitrogen is a suitable alloying element since it stabilizes the austenite in steel production and improves the strength at the same time [12, 13]. This characteristic is to be transferred for welding processing. The focus is on increasing the hardness of the weld metal in order to also infer the strength and on enhancing the impact strength. With regard to the microstructure, the objective is to continuously achieve an austenitic microstructure which does not exhibit any negative properties compared with the initial condition. In its entirety, it is envisaged to demonstrate that improved suitability for cryogenic applications can be realized in the processing of medium manganese austenites with basic filler materials as a result of nitrogen doping of the shielding gas.

## 2 Methods and materials

The investigations are based on the materials X2CrMnNi17-7-5 (1.4371) as the base material and G 20 16 3 Mn N L as the filler metal. The chemical compositions of both materials are listed in Table 1.

The material 1.4371 is a medium manganese austenite with low nickel level. There, the manganese content in particular serves to substitute the nickel and enables high strength and ductility. The plate thickness of the base material is 12 mm. The austenitic microstructure with apparent rolling structure is shown in Fig. 1. Regarding the impact energy at low temperatures, the measured values exceed 40 J/cm<sup>2</sup> at –196 °C. Consequently, the material is suitable for cryogenic applications. The filler metal is a standard wire processed in a wire diameter of 1.2 mm. The strength is approximately 650 MPa, and the impact energy is specified as at least 47 J/cm<sup>2</sup> at –196 °C. Both materials have a nitrogen content of about 0.18%.

### 2.1 Deductions obtained from preliminary studies on nitrogen doping

In preliminary tests, joint welds with 20° V-seam preparation and additional plate spacing of 12 mm have been carried out in accordance to DIN EN ISO 15792-1 (cf. Fig. 2) [14]. In addition, multilayer buildup welds have been performed. The energy per unit length is set to approximately 1.5 kJ/mm in compliance with the standards and guidelines of the

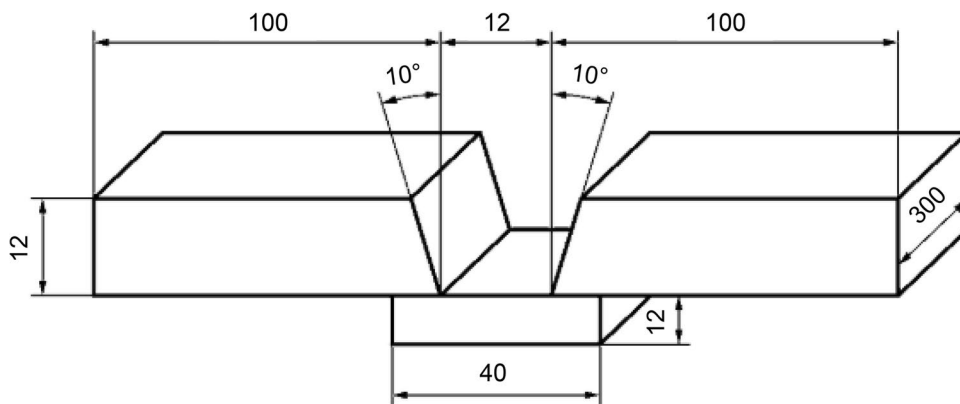


Fig. 1 Austenitic microstructure of the X2CrMnNi17-7-5 base material with obvious rolling structure

**Table 1** Chemical composition of base material and filler material

		Cr	Mn	Ni	C	Si	Mo	N	S	P
Base material	X2CrMnNi17-7-5 (1.4371)	16.71	6.52	4.50	0.03	0.54	–	0.18	<0.01	0.03
Filler metal	G 20 16 3 Mn N L	20.50	7.50	15.50	0.03	0.50	3.00	0.18	–	–

**Fig. 2** Schematic structure of the specimen with seam preparation according to [15]



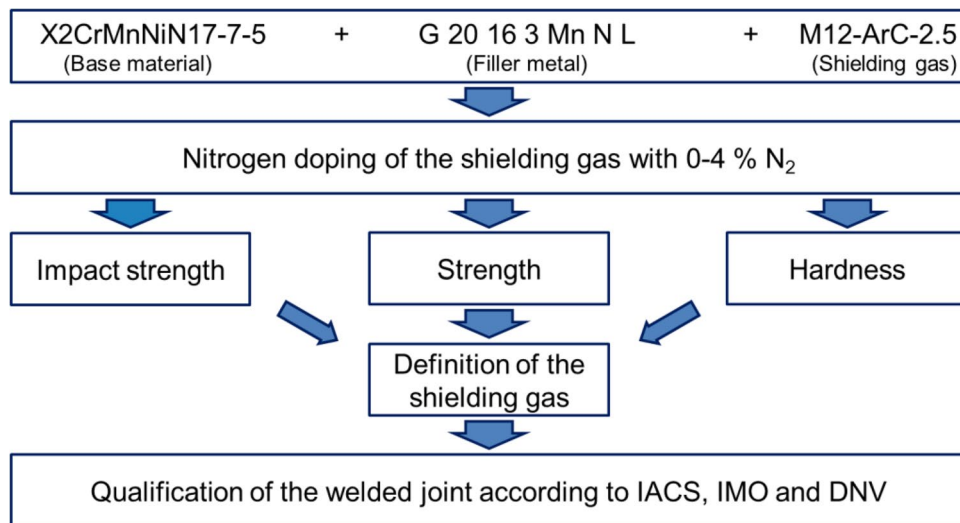
certification societies. The shielding gas M12, composed of argon and 2.5% CO<sub>2</sub>, is doped with nitrogen contents between 0 and 4%, and the weld metal is analyzed regarding the resulting mechanical properties. The proposed general procedure is shown in Fig. 3. By evaluating the impact energy, strength, and hardness resulting from different nitrogen contents, a definition of the shielding gas for the comprehensive qualification of the welded joint is made.

Based on the deposition-welded specimens, the nitrogen content was determined by means of carrier gas hot extraction of the pure weld metal. This serves to demonstrate the diffusion-based amount of nitrogen as a result of the welding process. In the initial state of the weld, about 2000 ppm N<sub>2</sub> can be detected in the weld metal. This is in line with the specifications of the filler metal, which state a nitrogen content of about 0.18%. As the nitrogen content in the shielding gas rises, there is an increase in the nitrogen content measured in the weld metal. In the case of doping with 4% nitrogen, the weld metal contains around 3300 ppm. Diffusion-based increase in the nitrogen content can therefore be demonstrated (cf. Fig. 4).

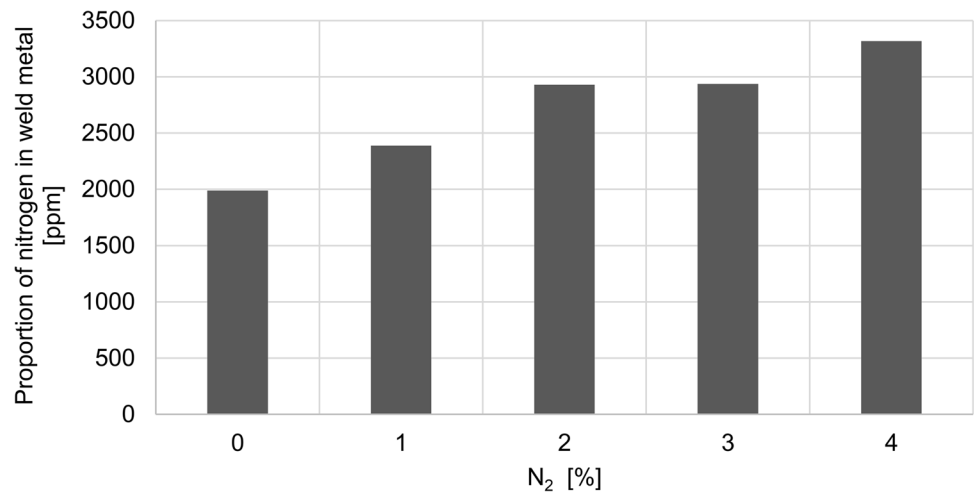
Based on the diffused nitrogen, significant influences on the impact energy at –196 °C and the tensile strength can be detected within the preliminary tests. While approximately 80 J/cm<sup>2</sup> respectively 590 MPa can be achieved in the initial condition, higher values can be obtained with increasing nitrogen content in the shielding gas. At 4% N<sub>2</sub>, an impact energy of 100 J/cm<sup>2</sup> and a strength of 640 MPa are realized (cf. Fig. 5). Thus, with regard to the mechanical properties, a clear influence of the nitrogen doping can be determined in the analysis. On the basis of these findings, the nitrogen content in the M12 shielding gas was set to 4% for the more extensive investigations. In addition, an M14 shielding gas with 5% as well as 6% nitrogen doping has also been included in the investigation due to requests from the industry. This was done against the background of the industry’s previous processing strategies.

It was found that the sheets show a tendency to significant deformation during welding of the V-weld preparation. As part of the qualification of the medium manganese austenite according to [3–5], the symmetrical K-weld preparation is used instead of a one-sided HV-weld preparation. The seam

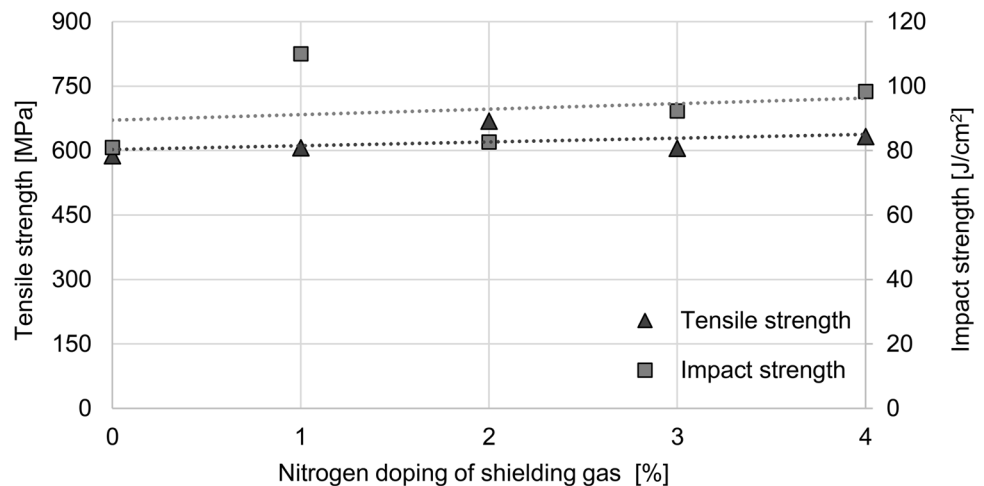
**Fig. 3** Schematic procedure for the selection of nitrogen doping in the shielding gas



**Fig. 4** Proportion of nitrogen in weld metal as a function of the nitrogen doping of the shielding gas



**Fig. 5** Tensile strength and impact strength of preliminary tests



opening angle is 45° in each case, and the seam preparation is applied to the sheet edges by milling. The torch is positioned at an angle of 10° to the weld center plane (cf. Fig. 6). The two welds are processed sequentially in a flat position (PA) while maintaining an interpass temperature of max. 90 °C. The energy per unit length is set to approximately 1.5 kJ/mm at a current of 290 A and a voltage of 26 V.

## 2.2 Applied analysis methods

For the determination of the ferrite content of the samples, the surface of the welded joint is scanned in a grid pattern using a FISCHERSCOPE MMS. This allows a specific location of the measured values to the characteristic areas of the weld seam and the base material. The metallographic examination of the welded joint is performed on a Leica DM6 M microscope system. The etching of the samples is carried out with V2A etchant (hydrochloric acid, nitric acid, and water at 50–70 °C) and Beraha II solution (hydrochloric acid, ammonium bifluoride, potassium metabisulfite, and

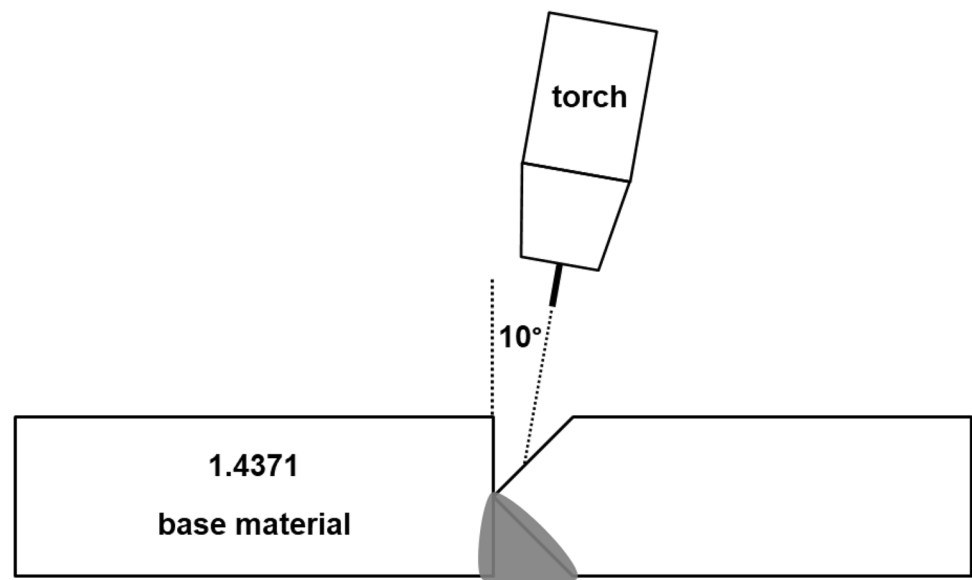
water at 20 °C). The different etchants allow first the visualization of the dendrites and furthermore also an exposure of the individual crystals. Vickers hardness measurements are carried out with a Struers DuraScan hardness tester. This setup allows automated hardness measurements with equidistant measuring points.

## 3 Results

### 3.1 Influence of nitrogen doping on the formation of the microstructure

The objective of nitrogen doping is the achievement of a microstructure which, as a result of the increased nitrogen content, does not exhibit any negative influences compared with the initial condition. In particular, this includes the realization of an austenitic microstructure with similar grain sizes compared to the application of undoped shielding gases. It was found that there were no significant differences

**Fig. 6** Schematic illustration of the experimental setup for the welding examinations

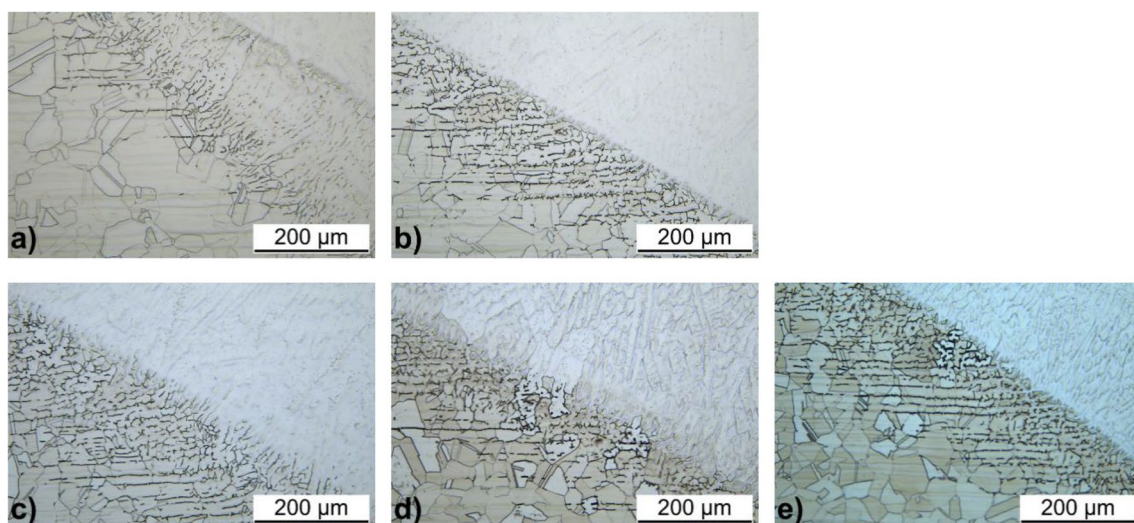


in the process during the welding of the material combination with the adapted shielding gases. In this regard, a metallographic examination of the resulting weld seams is carried out. Figure 7 shows sections of the joining zone of the welded joints of the two reference gases and the doped shielding gases. The specimens are etched with V2A etchant, which allows visualization of the grain boundaries. The austenitic base metal is clearly visible in the lower left section of the image, as is the solidification structure of the weld metal in the upper right section. Both areas are separated by a narrow-band joining zone.

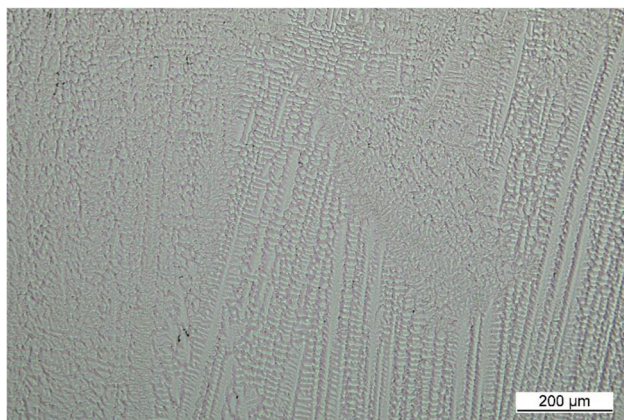
In the vicinity of the joining zone, precipitates can be detected with all five shielding gas variations. The width of the precipitates does not vary significantly between the

different samples. Basically, an alignment of the precipitates to the rolling orientation can be recognized. For the joining zone, no obvious difference between the welds can be detected on the basis of metallographic observation. The width of the area varies slightly depending on the depth within the weld. However, significant differences in width are not evident. The thermal influence on the microstructure of the base metal is negligible. For further metallographic analysis of the welds, the weld metal is considered in this regard.

Figure 8 shows a section of the weld metal of the joint generated using M12 shielding gas. The solidification structure with the column crystals is clearly visible. As expected, the formation of the column crystals occurs from the joining



**Fig. 7** V2A etching of the welded joints with base metal in the lower left image section and weld metal in the upper right image section; **a** M12 reference, **b** M12+4% N<sub>2</sub>, **c** M14 reference, **d** M14+5% N<sub>2</sub>, **e** M14+6% N<sub>2</sub>



**Fig. 8** V2A etching of the weld metal for M12 reference shielding gas

zone to the center of the weld. The microstructures of the different shielding gases show no obvious differences depending on the nitrogen doping. Using a DIC filter, the welded joints are examined again for depth information. Figure 9 shows the resulting microstructures, which exhibit clear three-dimensionality. The orientation of the column crystals stands out significantly and clearly shows crystal growth. Fundamental differences between the influences of the shielding gas doping on the microstructure, on the other hand, cannot be identified.

For the determination of the size of the individual crystals, color etching has been performed using Beraha II. The etching results in different colors due to the incidence of light and the orientation of the individual crystals, which allows a clear identification of the crystals. As a result, the respective crystals can be suitably distinguished from one another (cf. Fig. 10). However, no significant differences

in size can be determined. For all specimens, the expected crystal growth starting from the joint zone to the surface of the weld can be shown.

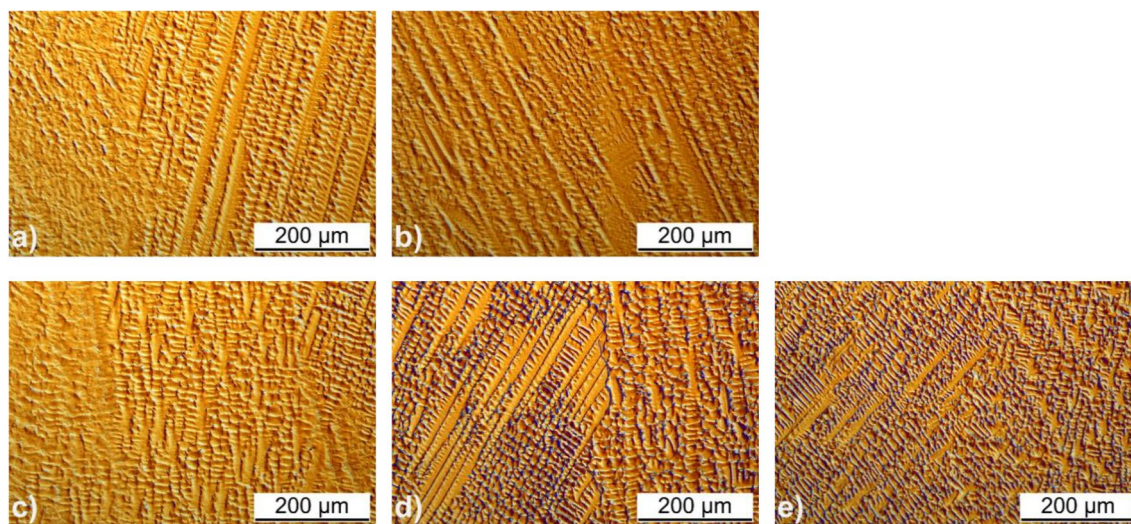
For a characterization of the microstructure, the ferrite content is examined in detail. Figure 11 shows the measured values of the ferrite content in the austenitic weld metal in relation to the shielding gas used. The welded joints of the M12 and M14 reference gases show ferrite contents of up to 2.5%. The average values are around 1.4 to 1.8%. In addition, significant scattering can be observed. Ferrite contents of around 0.5% can be achieved with the nitrogen-doped shielding gases. The measured values clearly demonstrate that a significantly higher ferrite content can be detected when using the undoped reference gases.

Whereas in the case of doped shielding gases, the ferrite content can only be measured in the joint zone (B, cf. Fig. 12), in the case of undoped shielding gas welds, the ferrite content can be measured over the entire weld (A, cf. Fig. 12). The measured values tend to be higher within the weld metal than in the joining zone. The influence of nitrogen on the austenite and ferrite content in the microstructure is consistent with the literature [10, 16].

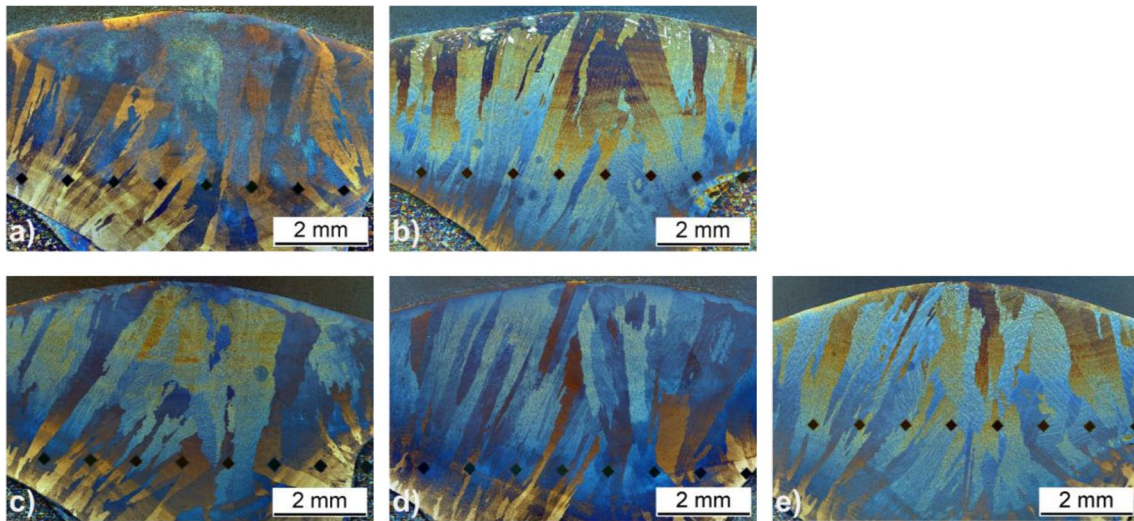
### 3.2 Nitrogen influence on the hardness of the weld metal

While the weld metal in its initial state does not match the hardness of the base metal, improvements are to be achieved as a result of nitrogen doping. The objective is a significant increase in hardness due to the diffused nitrogen.

The hardness of the welds was investigated in accordance with [5] at a distance of about 1.5 mm to the upper side of the sheet over the entire cross-section of the weld.

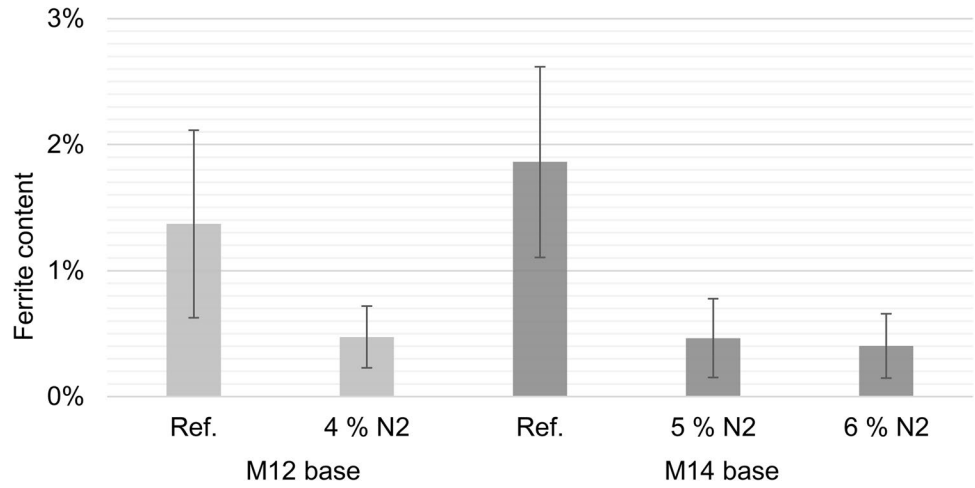


**Fig. 9** DIC-filtered V2A etching of the weld metal; **a** M12 reference, **b** M12+4% N<sub>2</sub>, **c** M14 reference, **d** M14+5% N<sub>2</sub>, **e** M14+6% N<sub>2</sub>

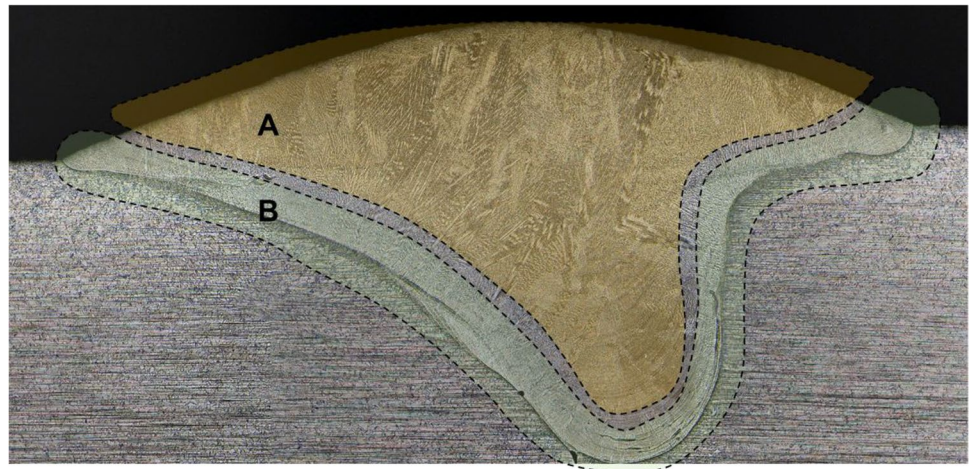


**Fig. 10** Beraha etching of the weld metal; **a** M12 reference, **b** M12+4% N<sub>2</sub>, **c** M14 reference, **d** M14+5% N<sub>2</sub>, **e** M14+6% N<sub>2</sub>

**Fig. 11** Measured ferrite contents in austenitic weld connection



**Fig. 12** Schematic overview of the area of detected ferrite content (**A** undoped shielding gases, **B** doped and undoped shielding gases)



The distance between the individual measuring points was chosen to be 1.00 mm (cf. Fig. 13). The resulting equidistant hardness measurements reveal that there is a slight reduction in the hardness values in the center of the weld metal (cf. Fig. 12). In the outer sections, the hardness reaches nearly the values of the base metal of about 210 to 230 HV10.

An average of the hardness measurements within the weld metal is calculated. Figure 14 shows the measured values as a function of the nitrogen content. In the undoped condition, the use of the M12 or M14 shielding gas results in a hardness in the weld metal of about 170 HV10. The difference between the two measured values is within the scatter band of the respective series of measurements. For the doped shielding gases, a significant increase in the attainable hardness within the weld metal can be detected. For the M12, a hardness of about 185 HV10 can be achieved with a nitrogen content of 4%. For the M14, over 180 HV10 can be achieved with 5% doping and over 185 HV10 with 6% N<sub>2</sub> doping.

A distinct correlation can thus be shown between the nitrogen content in the shielding gas and the hardness achieved. As a result, the hardness of the base material is nearly approached by nitrogen doping. With regard to the strength of the welded joint, an increase in strength can be assumed based on the measured hardness values.

### 3.3 Nitrogen influence on the impact strength of the weld metal

For the notched impact strength of the welded joints, the objective of nitrogen doping is to achieve increased impact strength compared to the initial state. The notch is located in the center of the weld metal. Figure 15 shows the resulting impact strength for measurements at -60 °C as well as at cryogenic conditions at -196 °C.

While the requirements according to standards and guidelines have been specified as a minimum of 40 J/cm<sup>2</sup>

Fig. 13 Positioning of measured hardness series for welded joints

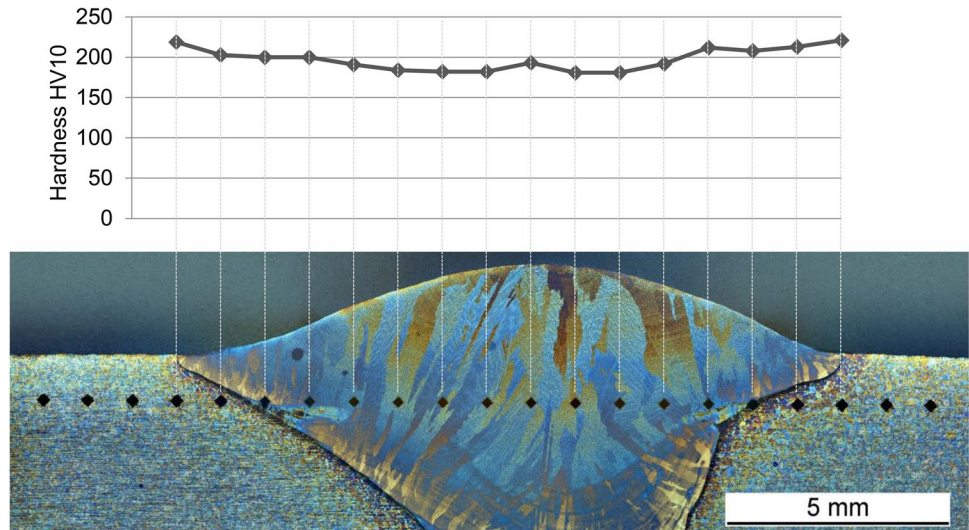
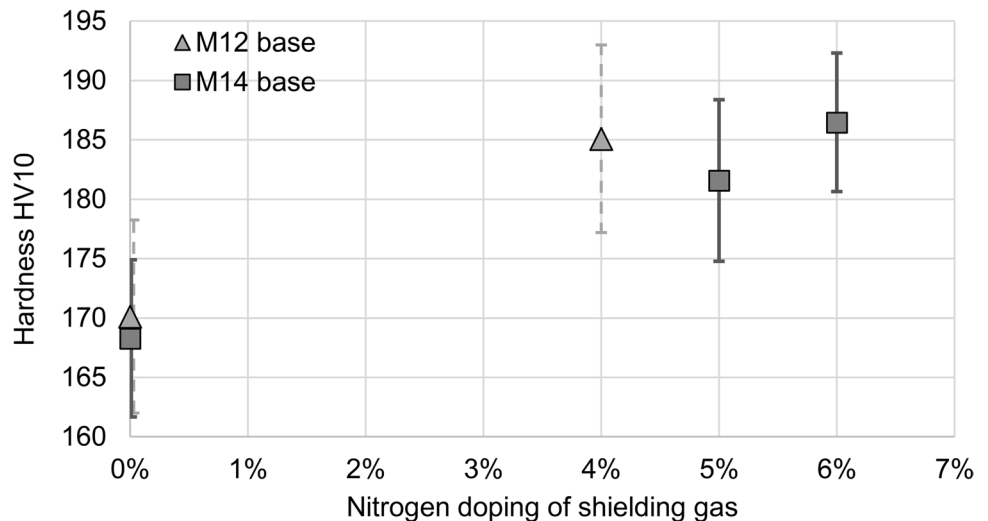
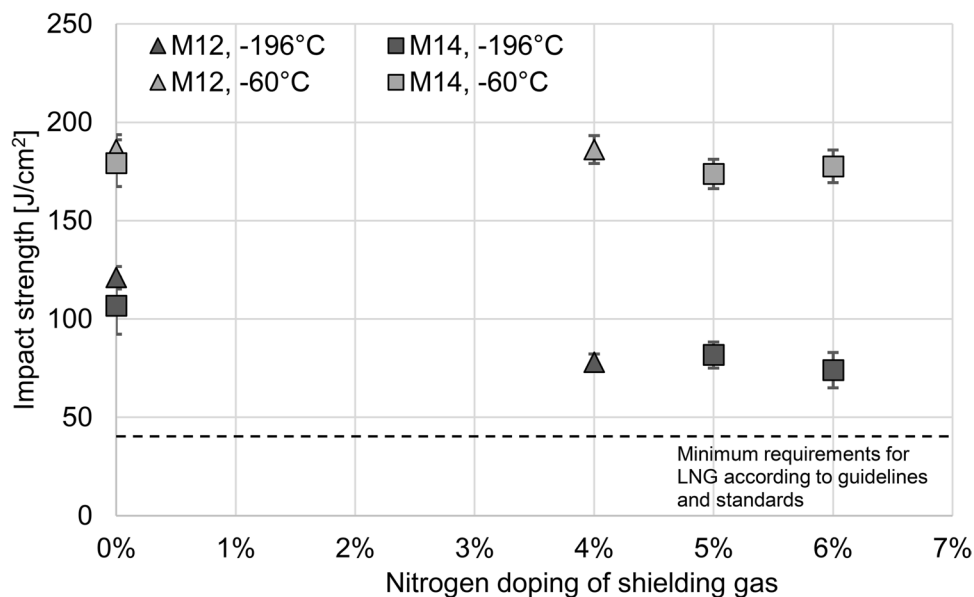


Fig. 14 Average hardness of the weld metal as a function of the nitrogen doping of the shielding gas





**Fig. 15** Average impact strength of the welded joints at  $-196\text{ }^{\circ}\text{C}$  and  $-60\text{ }^{\circ}\text{C}$  as a function of the nitrogen doping of the shielding gas



at  $-196\text{ }^{\circ}\text{C}$ , it can be determined from the measured values that these minimum requirements have been complied over all.

The impact strength of welded joints processed using M12 shielding gases remains nearly consistent at  $-60\text{ }^{\circ}\text{C}$ , increasing from about  $180\text{ J/cm}^2$  in the initial condition to about  $185\text{ J/cm}^2$  in the nitrogen-doped condition. At  $-196\text{ }^{\circ}\text{C}$ , in contrast, a significant reduction from approximately  $120$  to  $80\text{ J/cm}^2$  can be observed.

A similar characteristic can be identified for the M14 shielding gas variations. While at  $-60\text{ }^{\circ}\text{C}$ , an approximately constant impact strength of about  $170$  to  $175\text{ J/cm}^2$  is measured regardless of the nitrogen doping, at  $-196\text{ }^{\circ}\text{C}$ , these values decrease from about  $110$  to about  $80\text{ J/cm}^2$  at  $5\%$  and  $75\text{ J/cm}^2$  at  $6\%$  nitrogen doping, respectively.

The values presented do therefore not meet the requirements of the nitrogen doping objective. The doping does not seem to be accompanied by an increase in impact strength under cryogenic conditions. With reference to the preliminary investigations which showed a contrary behavior, upcoming investigations will clarify whether this characteristic can result from process irregularities with respect to the weld seam geometry.

#### 4 Summary and conclusions

Within the scope of the investigations, the influence of nitrogen doping of argon-based shielding gas has been considered for gas metal arc welding of an X2CrMnNiN17-7-5 (1.4371) medium manganese austenite base metal in combination with a G 20 16 3 Mn N L filler metal. Based on preliminary investigations, both shielding gases M12 and

M14 have been considered. The former is an adaptation with  $4\%$  nitrogen, the latter with both  $5\%$  and  $6\%$  nitrogen doping. The results of the preliminary investigations indicate a clear correlation between the doping of the shielding gas and the amount of nitrogen diffused into the weld metal.

While no significant optical differences are detectable in the metallographic examinations, and the microstructures of all welded joints are similar, differences can be identified by means of ferrite content measurements. The nitrogen content in the doped shielding gases results in significantly reduced ferrite contents within the welded joints. Moreover, while in the case of the undoped M12 and M14 shielding gases, the entire weld has a ferrite content of about  $1\text{--}2\%$ , only the joining zone is affected as a result of the nitrogen doping. Thus, most of the weld seam is entirely austenitic.

It can therefore be demonstrated that the austenite-stabilizing and austenite-initiating effect of the nitrogen is also achieved as a result of the doping of the shielding gas. The diffusing nitrogen thus induces a microstructural change, which, however, is not detectable within the metallographic micrographs. The slight difference in the ferrite content of the otherwise austenitic microstructure is not visible in the micrograph.

In addition, the diffusion-based increase in the nitrogen content in the welded joint leads to changes in the mechanical properties. If nitrogen is used in steels in particular as an alloying element to improve strength or surface hardening when nitriding to increase hardness, this can also be used for the welding of medium manganese austenites. The hardness of the welded joint is significantly increased as a result of nitrogen doping. This is exclusively due to the nitrogen diffused into the weld metal. Consequently, this allows the assumption that the strength of the welded joint can also be

significantly increased. This characteristic will be verified in further detail.

The impact strength at temperatures of about  $-60\text{ }^{\circ}\text{C}$  was not affected as a result of the nitrogen doping of the shielding gas. However, a significant reduction in the achievable values was observed in the cryogenic conditions. This behavior was contrary to the findings from the preliminary tests. One presumption suggests that, as a result of the different seam geometry, a process irregularity is the primary cause. Nevertheless, the obtained values clearly exceed the requirements from standards and guidelines for maritime applications.

It can be proven that the thesis and objective stated at the beginning of this paper can be fulfilled. The addition of nitrogen to the shielding gas has a distinct effect on the properties of the welded joint. Overall, it can be shown that there is no negative influence as a result of the doping besides the reduced impact strength. However, the requirements for cryogenic applications are still fulfilled.

The results demonstrate that doping of the shielding gas is also effective for this combination of materials. In summary, the following aspects can be listed:

- Nitrogen doping of shielding gas results in increased nitrogen content in weld metal for the material combination of X2CrMnNiN17-7-5 (1.4371) and G 20 16 3 Mn N L.
- Increased nitrogen level in the shielding gas leads to increased austenite formation and significantly reduced ferrite content.
- There is no optical microstructure change of the weld metal detected as a result of nitrogen doping.
- The hardness of the weld metal increases with higher nitrogen content.
- The impact strength does not increase as a result of nitrogen doping; under cryogenic conditions, the achievable values on the contrary decrease. The specifications for cryogenic applications are fulfilled nevertheless. An influence of process irregularity due to adapted weld geometry will be further investigated.

Based on the findings, further investigations of the shielding gas mixtures for welding applications will be analyzed under the given conditions. In particular, the extensive consideration of the qualification specifications for welded joints and filler materials of the standards and specifications according to IACS, IMO, and DNV [3–5] is in the foreground. In addition, a transfer of the findings on shielding gas doping to the processing of high manganese austenites is intended. Due to their higher manganese content, the properties of such materials exceed those of the material presented here.

**Funding** Open Access funding enabled and organized by Projekt DEAL. The research project IGF-Nr.: 22156 BG / FOSTA-Nr.: P 1597 Verarbeitung und Qualifizierung mittel- und hochmanganhaltiger austenitischer Stähle für die Lagerung kryogener Energieträger “ from the Research Association for steel Application (FOSTA), Düsseldorf, is supported by the Federal Ministry for Economic Affairs and Climate Action through the German Federation of Industrial Research Associations (AiF) as part of the program for promoting industrial cooperative research (IGF) on the basis of a decision by the German Bundestag. The project is carried out at the Fraunhofer Institute for Large Structures in Production Engineering IGP, Rostock, and the Institute of Welding and Machining at Clausthal University of Technology, Clausthal-Zellerfeld, Germany.

## Declarations

**Conflict of interest** The authors declare no competing interests.

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