Paper No.
11200

Alloy 699 XA - A new Alloy for Application under Metal Dusting Conditions

Heike Hattendorf
VDM Metals International GmbH
Kleffstrasse 23
D 58762 Altena
Germany

Alejandra López
Tubacex S.A.
Tres Cruces 8
01400 Llodio, Álava
Spain

Jutta Kloewer
VDM Metals International GmbH
Kleffstrasse 23
D 58762 Altena
Germany

ABSTRACT

Alloy 699 XA is a new alloy, which was developed for application in petrochemical industry under metal dusting conditions at high pressure. It contains 30% chromium and 2% aluminum. Due to this it showed a remarkable larger time to first pit in comparison to N06025 and N06690 in an examination in the highly carburization metal dusting gas 37% CO, 9% H_2O, 7% CO_2, 46% H_2, at 600 °C at 20 bar. Strength properties and creep properties are similar or better than N06601. Ductility at room temperature is comparable to N06601. The Charpy V-notch impact energy and the elongation in a tensile test at 600°C are still above the lower limits after aging solution annealed samples for 3000 h at 600°C. Welding procedure test according to DIN EN ISO 15614-1 was successfully done on 16 mm plate with a matching filler metal. Alloy 699 XA has been produced as plate, bar, seamless tube and wire.

Key words: Alloy 699 XA, metal dusting, creep strength, ductility at room temperature, V- notch impact energy, welding, plate, bar, tube, wire

INTRODUCTION

Metal dusting is observed in ammonia, methanol, hydrogen, and gas-to-liquids production plants. It is a high-temperature corrosion damage in iron, nickel or cobalt alloys, which are exposed to a carbon-bearing atmosphere with a carbon activity larger than one (i.e. mixtures of CO, hydrogen, water and CO_2) typically between 500 and 800°C.[1 - 7] CO from the gas atmosphere reacts at the metallic surface to form atomic carbon, which diffuses into the metal. The metal supersaturates in carbon and decomposes into a mixture of graphite, carbidic, oxidic, and metallic particles (“metal dust”).[8 - 24] The attack takes place by the formation of pits, but also a general attack is possible.[4 - 7]

The influence of the composition of wrought alloys has been studied in many papers.[9, 10, 13, 22, 25 - 28] The resistance to metal dusting is related to the ability of the alloy to form a protective oxide scale on its surface. Therefore high Cr, Al or Si content improve the resistance of an alloy against metal dusting. Additionally a low Fe content is of advantage.[28] Additions of Cu are reported to reduce the interaction of CO with the metal.[29 - 31] Furthermore there are influences of the grain size and the surface treatments such as machining, pickling, grinding [9 - 12, 32, 33, 34]

A metal used in such a carburizing atmosphere has not only to be sufficient resistant to metal dusting in this atmosphere, but e.g. has to have also a sufficient high creep strength and high-temperature strength. Based on these considerations the following targets for an alloy development were defined

- Higher metal dusting resistance than UNS N06025
- Creep strength at least like alloy UNS N06601
- Room temperature ductility better than UNS N06025 to produce seamless tubes
- Good weldability (under Argon)

In this paper, the properties of the newly developed metal dusting resistant alloy VDM[(1)] alloy 699 XA – alloy no. 2.4842 are presented.

EXPERIMENTAL PROCEDURE

Alloys and Production Routes

The nominal chemical compositions of the alloys, used in this investigation, are shown in Table 1. All compositions in this paper are in wt.-%.

Table 1
Nominal composition of tested alloys in weight percent

Alloy	UNS	Alloy No	Cr	Ni	Fe	Al	Others
600	N06600	2.4816	16	72	8	0.2	0.3 Ti
601	N06601	2.4851	23	60	14	1.4	0.4 Ti
690	N06690	2.4642	29	60	9	0.3	0.3 Ti
602 CA	N06025	2.4633	25	60	9	2	0.2 Ti, 0.06 Y, 0.08 Zr
699 XA	-	2.4842	30	Bal.	≤ 2.5	2	0.2 Nb, 0.05 Zr

UNS06600, UNS N06601, UNS N06025 and UNS N06690 are commercial alloy, which were chosen as reference alloys. The commercial heats for plate were hot rolled to final thickness and solution annealed. The commercial heat for strip was hot rolled to intermediate thickness, pickled and ground, then cold rolled and solution annealed. The commercial heat for bar was forged to final thickness and solution annealed. Alloy 699 XA was melted in an electrical furnace and electro slag remelted (ESR). Then hot rolled and solution annealed plate, hot and cold rolled and solution annealed plate, wire and rods for welding and hot rolled and solution annealed bars were produced. Additionally, a bar was forged and solution annealed to perform the various tests that will be described below, and a billet was forged as prematerial for tube production. From this billet tubes were made by hot extrusion and subsequent cold pilgering by Tubacex[(2)]. Also laboratory heats of about 10 kg from alloy 699 XA were

[(1)] Trade name
[(2)] Company name

examined. These were melted in a vacuum furnace, then hot formed to final thickness and solution annealed. All materials, with exception of the tubes, were produced by VDM Metals[(3)]. From the materials suitable blanks for the different tests were cut.

Testing procedures

The investigations performed for this study are presented in the following, accompanied with the results, discussion and comparison with literature.

RESULTS

Metal Dusting Corrosion Tests

The samples listed in Table 2 were tested in a metal dusting corrosion test at TNO[(4)].

Table 2
Grain size and sample thickness of the tested materials

Code	Code old	UNS	Alloy	Lab heat	Heat	Product	Grain size in µm	Sample thickness in mm
T18		N06601	NiCr23Al1		165306	strip	13	4.45
T24		N06601	NiCr23Al1		165306	strip	267	4.49
T11	153	N06690	NiCr29		131986	plate	*62*	4,12
T21		N06690	NiCr30		321770	bar	96	5.00
T01	150	N06025	NiCr25Al2		155202	plate	85	4.55
T04	152	N06025	NiCr25Al2		156825	strip	34	1.83
T09		-	NiCr28Al2Fe0.5	x	250138	plate	234	3.95
T08		-	NiCr30Al2Fe0.5	x	2301	plate	162	3.98

From the blanks 4 to 6 mm thick test coupons were machined. From heat 156825 2 mm thick test coupons were machined. The surfaces of all samples were ground down to 600 grit by TNO. The final dimensions of the samples were about 50 mm x 10 mm x thickness. (See Table 2) Samples from the same lot of UNS N06025 were already examined in another examination at TNO.[22, 34] Determination of the grain sizes in Table 2 was done by linear intercept method.

For the metal dusting tests a high pressure high temperature setup as described already in a previous paper[21, 22] was used to perform the exposure. A temperature of 600°C was chosen, since at this temperature the metal dusting attack is most severe. The high pressure was chosen to be near the industrial conditions. This is critical, since previous works show that the total pressure has a significant effect on the severity of the metal dusting attack.[21, 35]

Long-term exposures were run at 600°C and 20 bar using the so-called gas 2B. This is a gas with a high carbon activity gas used by TNO to rank very high metal dusting resistant alloys. The composition of gas 2B is listed in Table 3 together with the activities calculated for the CO reduction reaction, the Boudouard reaction and at water gas shift equilibrium. After each exposure of about 125 h, the samples were cleaned ultrasonically, examined for pits and photos were taken.

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(3) Company name
(4) Netherlands Organization for Applied Scientific Research (TNO), PO Box 6235, 5600HE Eindhoven, NL.

Table 3
Gas composition, oxygen partial pressure and carbon activity at 600°C and 20 bar pressure

Gas	Composition, vol.-%				pO_2, bar	Carbon activity a_c		
	CO	H_2O	CO_2	H_2		CO reduction	Boudouard	at water gas shift eq.
2B	37	9	7	46	$2.56\ 10^{-27}$	163	452	253

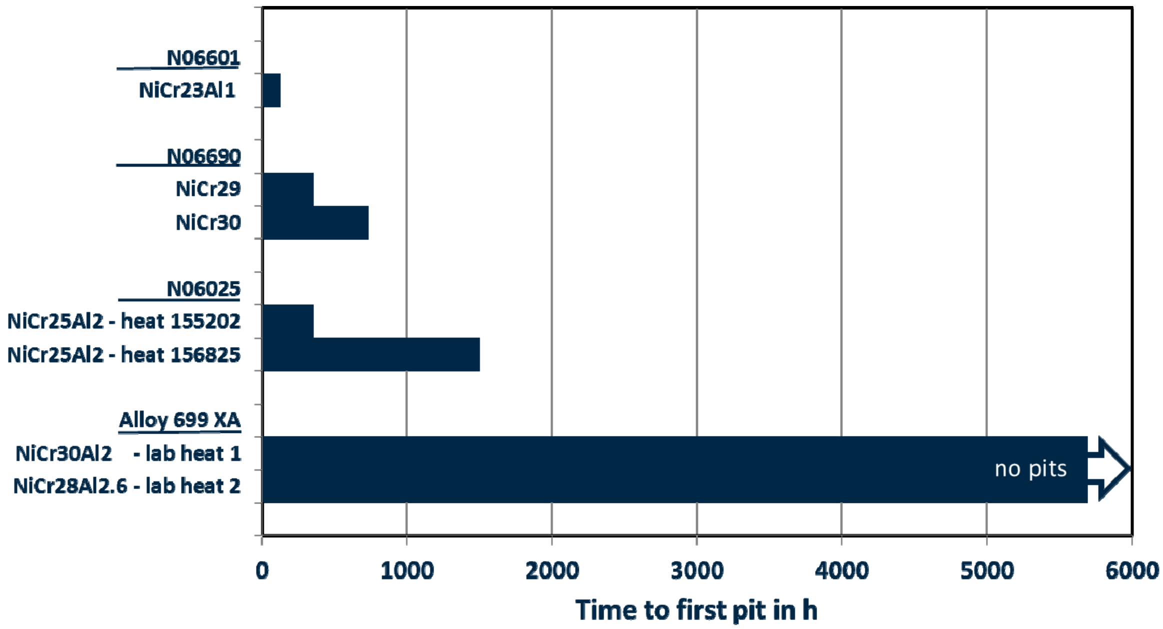


Figure 1: Time to first pit (no matter where).[36, 37]

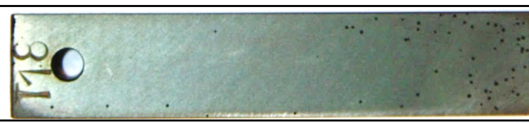
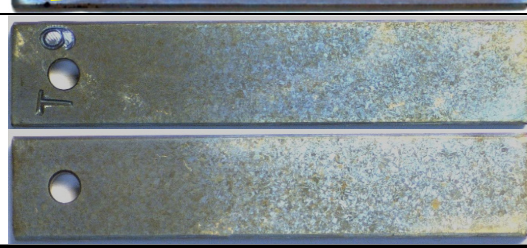
Alloy	Photos after	Photo	Pits
N06601 NiCr23Al1	123 h		many pits
N06690 NiCr30	754 h		1 pit
N06025 NiC25Al2 heat 156825	1499 h		few pits
Alloy 699 XA NiCr30 Al2	5693 h		no pits on all sides only surface discolouration

Figure 2: Photos after appearance of the first pit or end of exposure respectively.[36, 37]

Figure 1 shows the time to first pit no matter, where it is on a surface or an edge for the different materials and Figure 2 shows photos of a samples of each alloy after showing the first pits or at the end of exposure time respectively. [36, 37] The 2 samples of N06601 show many pits after the first break at 123 h. Then up to about 1500 h the different heats of N06690 and N06025 follow showing few pits, starting with NiCr30 (UNS 06690) and heat 155202 (UNS 06025). The samples from heat 156825 (UNS 06025) show the longest time of about 1500 h for the UNS 06025 and UNS 06690 samples. The different behavior concerning the times to first pit for the 2 heats of UNS 06025 was already observed in [34] in a different furnace run. Alloy 699 XA shows no pit and no attack on edges until 5693 h, when the test had to be terminated, because of the close down of the site of the TNO in Apeldoorn. Only surface coloration can be seen. Alloy 699 XA therefore shows a remarkable increased resistance against metal dusting in comparison to alloy N06601 and N06025. For a more detailed description of the results see the reference [37].

Phase diagrams

Phase calculations were performed with JMatPro V9.0[(5)] on alloy 699 XA. Figure 3 shows the phase diagram in the thermodynamical equilibrium for the composition 68% Ni, 2.0% Al, 30% Cr, 0.5% Fe, 0.15% Nb, 0.01% Zr, 0.026% N and 0.023% C. The following phases are predicted: liquid, austenitic gamma γ, gamma prime γ' ($(Ni,Ti)_3Al$) below 755 °C, carbides $M_{23}C_6$ below 1165°C and nitrides (MN). The calculated liquidus temperature is 1388°C. The calculated solidus temperature is 1350°C.

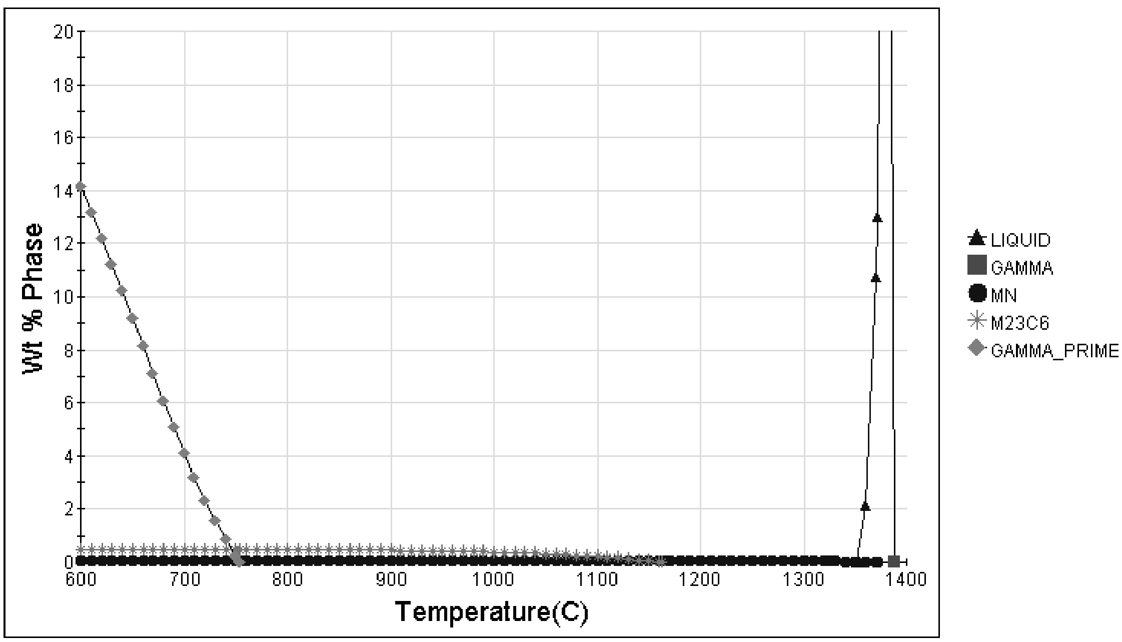


Figure 3: Phase diagram of alloy 699 XA calculated with JMatPro V9.0

(5) JMatPro the Materials Properties Simulation Package Version 9.0, Sente Software Ltd. Surrey Technology Centre 40 Occam Road GU2 7YG United Kingdom

Creep Tests

Non-interrupted uniaxial creep tests in tension with strain measurement according to DIN EN ISO(6)204[38] were done on solution annealed samples from a forged bar for alloy 699 XA at SZMF(7) and MPA/IFW(8) Creep tests on annealed tubes of alloy 699 XA were done in Tecnalia[9]. For the reference alloys N06601 and N06600 interrupted creep tests according to DIN 50118[39] were carried out at Metal-Laboratory(10) and SZMF on samples from solution annealed hot rolled plate. For 699 XA creep data[40] between 550°C to 950°C in steps of 50°C up to 8500 hours on one heat were available. For N06600 data[42, 43] at 550°C, 650°C and 850°C up to 40000 hours on 2 heats and for N06601 data[44-50] at 600°C, 620°C, 650°C, 700°C, 750°C, 850°C, 900°C and 1100°C up to 38500 hours on 4 heats were available.

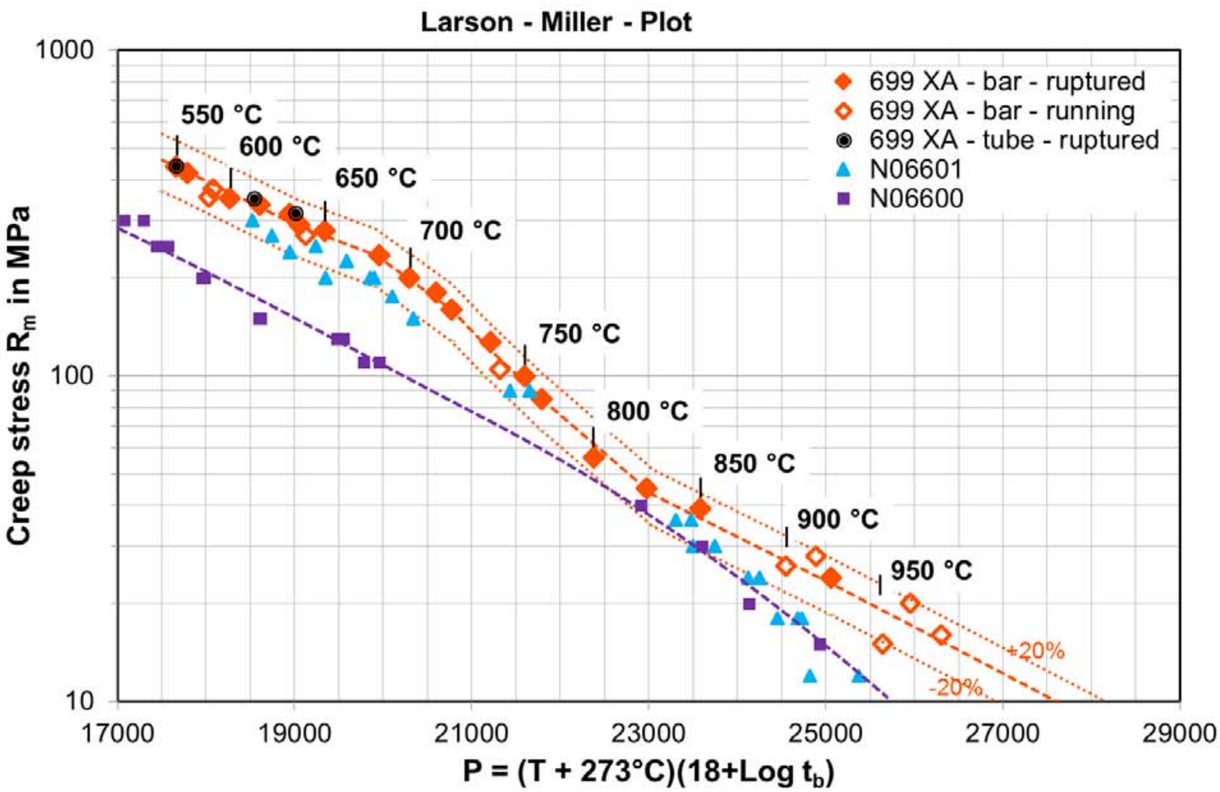


Figure 4: Larson-Miller-Plot of the creep results of alloy 699 XA on bar[40, 41], alloy 699 XA on tube, N06600[42, 43] and N06601[44 -50] T: test temperature in °C, t_b: time to rupture in hours.

The data on alloy 699 XA, N06601 and N06600 were analyzed after Larson-Miller.[51] The best fit was achieved for C = 18 in the parameter

$P = (T + 273°C)(C + \text{Log } t_b)$ with $\mathbf{t_b}$: times to rupture in hours, T: temperature in °C

(6) DIN EN ISO: DIN Deutsches Institut für Normung e. V., Am DIN-Platz, Burggrafenstraße 6, 10787 Berlin; EN European Standard; ISO International Organization for Standardization
(7) Salzgitter Mannesmann Research Institute (SZMF), PO Box 47251 Duisburg, Germany
(8) MPA/IFW Center for Structural Materials State Materials Testing Institute Darmstadt (MPA) - Institute for Materials Technology (IfW) Grafenstraße 2, 64283 Darmstadt, Germany
(9) Tecnalia E-20009 Donostia-San Sebastián - Gipuzkoa (Spain)
(10) Metal-Laboratory, Metallgesellschaft A.G. Frankfurt, Germany

A plot of creep stress over P is shown in the Larson-Miller-Plot in Figure 4 for all 3 alloys. For 699 XA the lines for the mean value and mean value - 20% and + 20% are drawn. The curve of alloy 699 XA in the Larson-Miller plot is less steep at temperatures below 700°C and above 800°C. The high creep strength in the temperature range between 550 to 750°C is caused by γ' phase precipitates ($(Ni,Ti)_3Al$), as shown by the calculations with JMatPro in Figure 3. Figure 4 shows, that the goal "creep strength at least like alloy UNS N06601" has been achieved.

Tensile tests at room temperature

To investigate room temperature ductility results of several hundred tensile tests at room temperature according to DIN EN ISO 6892-1[52] performed after production of N06601 and N06025 both on solution annealed hot formed plate were collected. For alloy 699 XA tensile tests at room temperature according to DIN EN ISO 6892-1[52]on bar and plate at VDM and on tube at Tubacex were performed. All samples were solution annealed. The ranges of the results are shown in Table 4.

As was to be expected from the composition the yield and tensile strength was the highest for N06025 and the lowest for N06601 with alloy 699 XA in between. N06025 showed the lowest elongation from 30 to 54%, N06601 a high elongation of 44 to 68%. Alloy 699 XA has an elongation in the same range of about 60%. The goal "room temperature ductility better than UNS N06025" was achieved.

Table 4:
Results of room tensile test on Alloys N06601, N06025 and alloy 699 XA

Alloy	Yield Strength $R_{P0.2}$ in MPa	Tensile strength R_m in MPA	Elongation A_5 in %
Alloy 699 XA (from first mother melt)	255 - 390	650 - 750	47 - 68
N06601	220 - 300	550 - 710	44 - 68
N06025	270 - 390	675 - 780	30 - 54

Hot tensile tests

Also hot tensile tests up to 850°C according to DIN EN ISO 6892-2[53] were performed on alloy 699 XA bar at VDM and SZMF[40] and on tube at Tubacex. All samples were solution annealed. The results for the yield and the tensile strength and the elongation are shown in Figure 5.

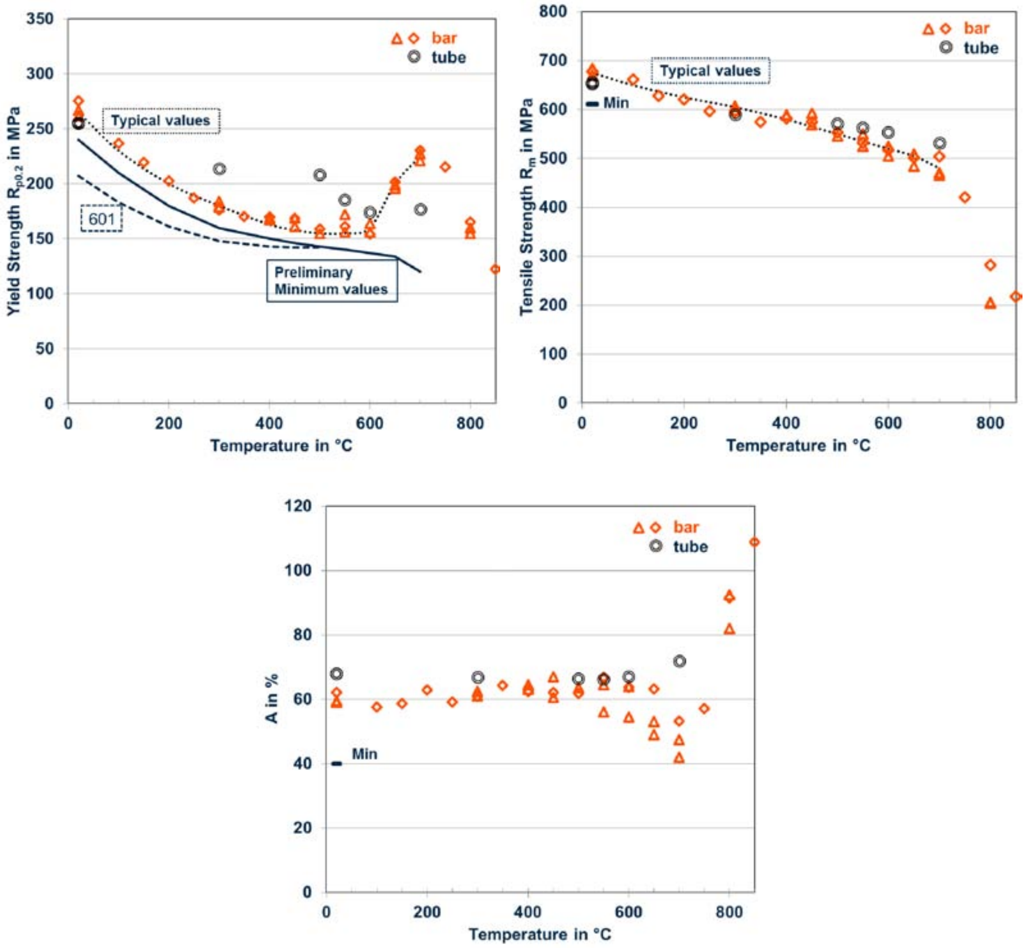


Figure 5: Yield and tensile strength and elongation of hot tensile tests on alloy 699 XA bar and tube (solution annealed), triangle measured by [40]

In Figure 5 above left, the minimum values for the yield strength for N06601 according to[54] and the preliminary minimum values for alloy 699 XA are also added (see also Table 5 for the values). The minimum values for alloy 699 XA are higher (at room temperature) to equal (at about 500 to 600°C) those of N06601. Furthermore, the preliminary minimum values for the tensile strength and the elongation are added to Figure 5 above right and below left respectively. All measured values on alloy 699 XA bar and tube are well above the minimum values.

Table 5
Preliminary minimum values for the yield strength in dependence of temperature, the tensile strength and the elongation at room temperature

T in °C	20	100	200	300	400	500	600	700
$R_{p0.2}$ in MPa	240	210	180	160	150	143	137	120
R_m in MPa	610	-	-	-	-	-	-	-
A in %	40	-	-	-	-	-	-	-

Charpy V-notch bar impact test

Also Charpy V-notch bar impact tests at room temperature according to DIN EN ISO 148-1[55] were performed on alloy 699 XA bar, plate and tube. All samples were solution annealed and had the dimension 10 mm x 10 mm x 55 mm with V-notch. One value is the average value of the tests on 3 samples. Figure 6 shows a histogram of the results.

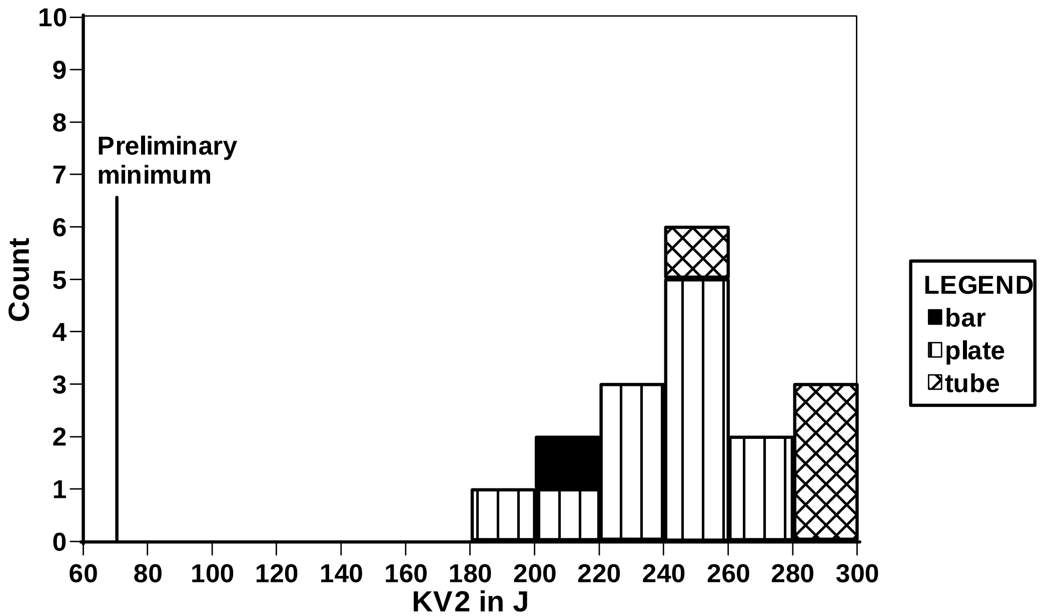


Figure 6: Histogram of the V-notch bar impact energy KV_2 at room temperature on alloy 699 XA bar, plate and tube.

The results are well above the preliminary minimum value of 699 XA of 70 J (See Table 6).

Table 6
Preliminary minimum value of V-notch bar impact energy at room temperature on alloy 699 XA. For comparison, the value for N06025 is added.

Alloy	KV_2 in J
699 XA	≥ 70
N06025	≥ 55

Results after Aging (on bar)

Blanks from bar for V-notch bar impact tests and tensile test at room temperature (RT) were aged at 600°C for 500 h, 1000 h and 3000 h. After the aging the tests were performed. Figure 7 shows the results. The impact energy KV_2 after annealing at 600°C is reduced, but remained above 100 J. This is well above the lower limit of 70 J after solution annealing. The yield strength increases from values of about 20 MPa to about 450 MPA, and the elongation is reduced, but still above 45 % after 3000 h.

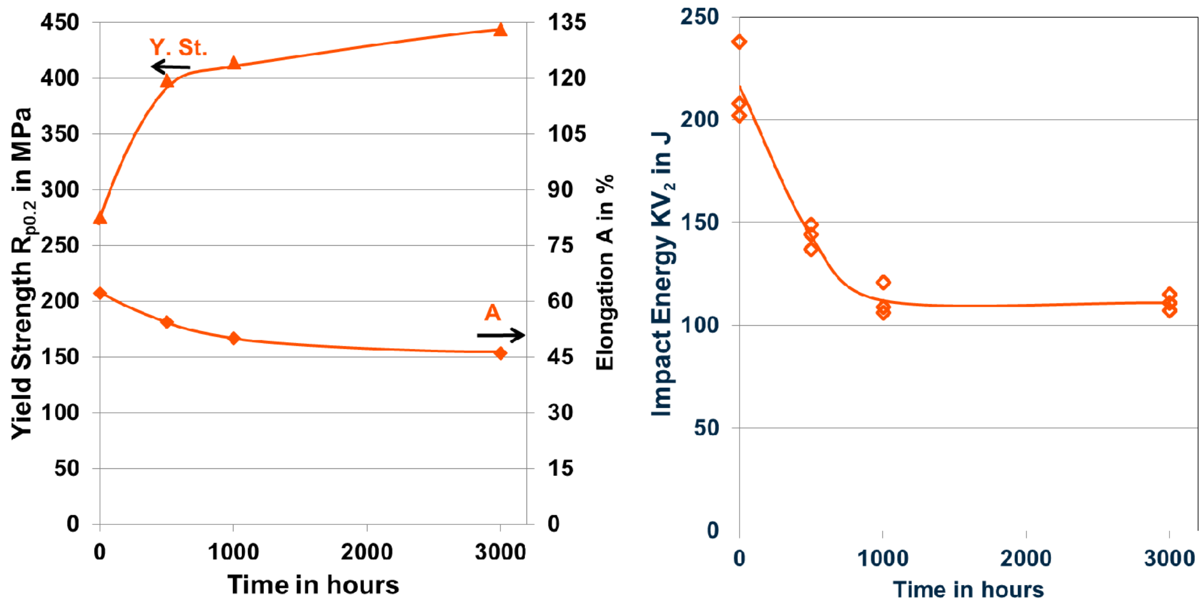


Figure 7: Yield strength and elongation (left) and Charpy V-notch impact energy after annealing solution annealed samples of alloy 699 XA, heat 318385 - bar up to 3000 h at 600°C.

Welding

Plates of alloy 699 XA of 16 mm thickness were welded with gas tungsten arc welding method (GTAW) with 2.0 and 2.4 mm rod under argon using matching filler metal. Afterwards a welding procedure test according to DIN EN ISO 15614-1[56] was performed successfully. This includes visual inspection, a liquid penetrant examination, a radiographic test, a hardness test, metallographic examination, V notch impact bar tests, a tensile test at room temperature and bend over tests all perpendicular to the weld, which were all passed well.[57] Figure 8 shows a macro picture of the weld and Figure 9 a metallographic picture of the weld and the heat-affected zone.

The goal “good weldability under Argon” was achieved.

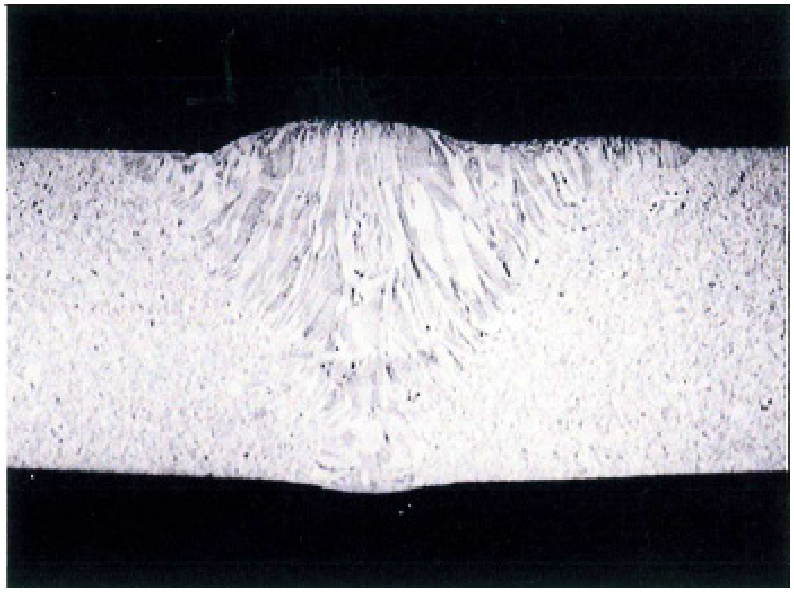


Figure 8: Macroscopic picture of the weld[57]

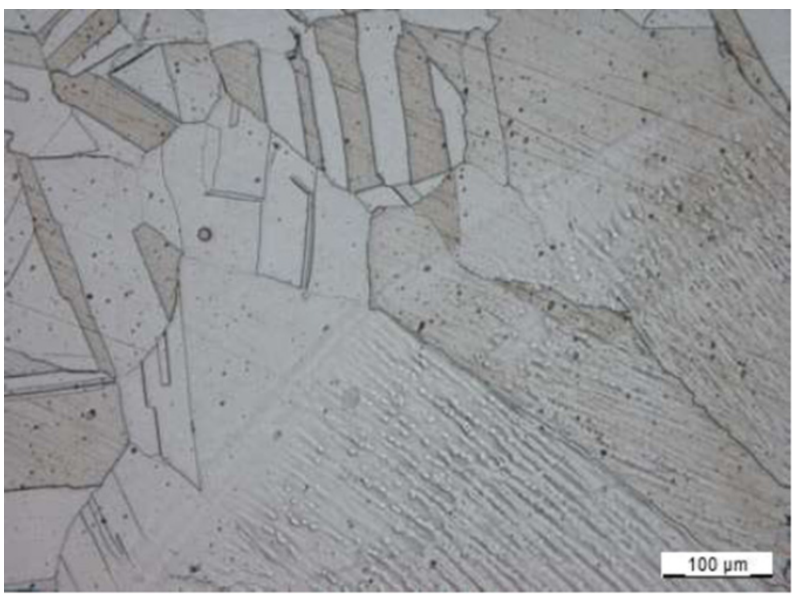


Figure 9: Metallographic picture of the weld and the heat affected zone.

CONCLUSIONS

- Alloy 699 XA contains 30% chromium and 2% aluminum and is highly resistant against metal dusting. It showed a remarkable larger time to first pit in comparison to N06025 and alloy N06690.
- Hot strength properties and creep properties of alloy 699 XA are similar to or better than N06601.
- Ductility at room temperature of alloy 699 XA is comparable to N06601.
- The impact energy and the elongation in a tensile test at 600°C are still above the lower limits after aging solution annealed samples for 3000 h at 600°C.
- The weldability is good. A welding procedure test according to DIN EN ISO 15614-1 successfully done with 16 mm plate.
- Hot and cold rolled plate, welding wire and rods, forged bars and billets, rolled bars and seamless tubes were successfully produced.

ACKNOWLEDGEMENTS

The authors wish to thank R. IJzerman, J.-P. Krugers from TNO, Chrétien Hermse former TNO now Shell, Stefanie Siegfanz, Paul Neddermann, from Salzgitter Mannesmann Research Institute, Dr. Martin Wolf, Johannes-Peter Kemper, Gabriele Toth and Michael Hebgen from VDM Metals for their contribution on to this work.

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