# Nicrofer<sup>®</sup> 4320 Ti

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# **Corrosion-resistant alloy**

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A company of ThyssenKrupp Stainless



# Nicrofer® 4320 Ti

Nicrofer 4320 Ti is a  $\gamma$ '-phase precipitation-hardenable austenitic nickel-iron-chromium alloy containing molybdenum, copper, titanium and aluminium. It exhibits high strength at temperatures up to approx. 1020 °F (550 °C) and shows excellent corrosion resistance including resistance to sulfide stress cracking (SSC) under  $H_2S$  containing sour gas conditions. It is used for surface and down-hole applications in sour gas wells and in oil production.

#### **Designations and standards**

Country	Material designation	Specification									
National standards	5	Chemical composition	Tube ar seamless	nd pipe welded	Sheet and plate	Rod and bar	Strip	Forgings	Wire		
D DIN VdTÜV	<b>WNr. 2.4852</b> NiCr20FeMo3TiCuAl										
F AFNOR											
UK BS											
<b>USA</b> ASTM	UNS N09925										
ASME ASME Code Case				2218 (Sec.	VIII, Div. 1)						
NACE	Material listed in MR 0175										
ISO											

Table 1 – Designations and standards.

#### **Chemical composition**

	Ni	Cr	Fe	С	Mn	Si	Мо	Cu	Ti	AI	Nb	Р	S
min.	42.0	19.5	22.0				2.5	1.5	1.9	0.1			
max.	46.0	22.5		0.03	1.0	0.5	3.5	3.0	2.4	0.5	0.5	0.03	0.03

Table 2 – Chemical composition (wt.-%).

#### Physical properties

Density	8.1 g/cm <sup>3</sup>	0.292 lb/in.3		
Melting range	1311 – 1366 °C	2392–2490 °F		
Permeability at 20 °C / 68 °F at 200 Oersteds (15.9 kA/m)	1.001			

Temperat	ure (T)	Specific he	eat	Thermal conductivit	у	Electrical resistivity		Modulus of elasticity	:	Coefficient thermal ex between room temp and T	of pansion perature
°C	°F	 kg K	<u>Btu</u> Ib °F	W m K	Btu in. ft² h °F	$\mu\Omega$ cm	$\frac{\Omega \text{ circ mil}}{\text{ft}}$	<u>kN</u> mm <sup>2</sup>	10 <sup>3</sup> ksi	<u>10<sup>-6</sup></u> K	<u>10<sup>-6</sup></u> °F
20	68	435	0.104	12.0	83	117	70	199	28.9		
93	200		0.109					195	28.3		7.8
100	212	456		12.9	89					13.2	
200	392	486		14.3	99					14.2	
204	400	485	0.116					188	27.3	14.6	8.1
300	572	507		15.9	110					14.7	
316	600		0.122					182	26.4	14.9	8.4
400	752	532		17.4	121					15.0	
427	800	540	0.129					175	25.4	15.2	8.5
500	932	561		19.3	134					15.3	
538	1000		0.136					168	24.4		8.7
600	1112	586		22.2	154					15.7	
649	1200	598	0.143					160	23.2		9.0
700	1292	611		24.0	167					16.3	
760	1400		0.150					150	21.8		9.5
800	1472	641		28.2	196					17.2	
871	1600	657	0.157					139	20.2		
900	1652	666		27.2	192						

Table 3 – Typical physical properties at room and elevated temperatures.

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#### **Mechanical properties**

The following properties are applicable to Nicrofer 4320 Ti in the solution-annealed and age-hardened condition and indicated size range.

Product	Dimensions thickness/dia mm	ameter inches		Tensile s R <sub>m</sub> N/mm²	trength ksi	0.2 % Yie R <sub>p0.2</sub> N/mm²	ld strength ksi	1.0 % Yie R <sub>p1.0</sub> N/mm²	ld strength ksi	Elongation A <sub>5</sub> %	Rockwell hardness* HRC
11.1.1.1.1.			Average	1133	164.3	810	117.5	868	125.9	28.5	32.6
forged Rod & bar	≤ 225	≤ 8.75	Range	1092 to 1188	158.4 to 172.3	734 to 898	106.5 to 130.2	805 to 950	116.8 to 137.8	27 to 30	31 to 36

\*Acceptable in the solution-annealed and aged condition to 35 HRC maximum according to NACE Standard MR0175

Table 4 – Average and range of mechanical properties at room temperature. in the solution-annealed and TK VDM two stage precipitation heat treated condition.

#### ISO V-notch impact toughness

Average values at room temperature:  $\geq$  110 J/cm<sup>2</sup>

#### Slow strain rate test results

Slow strain rate tests in various sour environments using a strain rate of  $4 \times 10^{-6} \text{ s}^{-1}$  corresponding to a crosshead displacement rate of  $1.02 \times 10^{-4}$  mm s<sup>-1</sup> yielded results as shown in Table 5. In all cases round tensile samples were machined according to NACE Standard TM 0177, Practice A and electropolished to minimize the influence of cold work on the surface as a result of machining.

#### Metallurgical structure

Nicrofer 4320 Ti is a precipitation-hardenable austenitic Ni-Fe-Cr alloy with additions of titanium and aluminium. Precipitationhardening, which is carried out in a dual step thermal treatment to ensure only formation of  $\gamma$ '-phase and to avoid particularly formation of brittle phases such as  $\sigma$ -phase, considerably increases the alloy's hardness and tensile strength. Prolonged exposure to higher temperatures causes detrimental phases to form. Fig. 1 shows the time-temperature-transformation (TTT) diagram for Nicrofer 4320 Ti material in the initially solution-annealed condition.



Fig. 1 – Time-temperature-transformation diagram for solutionannealed Nicrofer 4320 Ti material.



Fig. 2 – Typical microstructure of solution-annealed Nicrofer 4320 Ti 214 mm (8.5 in.) diameter rod.

Medium	Temperature	Final fracture load	Time of failure		Reduction of area		
		kN	h	Ratio (%)	%	Ratio (%)	
А	RT	32.0	21.08		38.7		
В	177 °C (350 °F)	32.8 32.6 33.8	23.17 20.28 20.37	110 96.2 96.6	28.6 25.9 27.7	73.9 66.9 71.6	
С	RT	32.1	21.98		47.9		
D	149 °C (300 °F)	31.4 33.3 31.9	17.08 19.77 23.05	77.7 89.9 104.9	33.2 29.7 33.6	69.3 62.0 70.1	

Medium A: 0.69 MPa N2 deionised water

Medium B: 25 % NaCl, 0.5 % acetic acid, 0.69 MPa H\_2S

Medium C: 5 MPa N2 deionised water

Medium D: 25 % NaCl, 0.172 % MPa H<sub>2</sub>S, 4.83 MPa CO<sub>2</sub>

Table 5 – Slow strain rate test results in various sour environments.

#### **Corrosion resistance**

The high chromium content as well as molybdenum and copper contents make Nicrofer 4320 Ti material very resistant to all forms of corrosion in a variety of chemical media under both reducing and oxidizing conditions. Due to its high nickel content Nicrofer 4320 Ti also exhibits virtual immunity to chloride-induced stress-corrosion cracking. Particularly useful is Nicrofer 4320 Ti no il and sour gas environments as well as seawater.

Critical pitting and crevice corrosion temperatures (CPT and CCT) of Nicrofer 4320 Ti determined in accordance to ASTM G48, Methods C and D respectively, indicated the respective values to be similar for both materials in the solution-annealed as well as in the solution-annealed and age-hardened condition as shown in Fig. 3.

Table 6 lists CPT and CCT together with Pitting Resistance Equivalent (PRE) values for Nicrofer 4320 Ti and various other similar alloys for comparison.





## Nicrofer® 4320 Ti

Alloy	UNS	CPT* in °C	CCT* in °C	PRE**			
Nicrofer 4320 Ti	N 09925	30	< 10	29			
Alloy 825	N 08825	30	< 5	33			
Alloy 625	N 06625	77.5	57.5	51			
Alloy 926	N 08926	72.5	47.5	47			
Alloy 31	N 08031	85	65	54			
*Determined according to ASTM G48 Method C and D respectively (10% Fe Cl <sub>3</sub> · $6H_2O$ ; 2.5 °C temperature steps up to 85 °C max.)							

Table 6 – Comparison of CPT, CCT and PRE values for Nicrofer 4320 Ti and other alloys used in the oil and gas industry.

C-Ring tests performed according to NACE Standard TM 0177, Method C in various test solutions and different periods of exposure varying from 30 to 61 days showed no sensitivity

for Nicrofer 4320 Ti to stress-corrosion-cracking (SCC) in H<sub>2</sub>S environments. Details of the tests are shown in Table 7.

Material condition	Test medium	Temperature	0.2 % Yiel R <sub>p0.2</sub> N/mm <sup>2</sup>	d strength ksi	Applied stress % YS	Test duration days	SCC
age-hardened* age-hardened*	A	RT	822 753	119.2 109.2	100	30	no no
age-hardened* age-hardened*	В	RT	822 753	119.2 109.2	100	30	no no
age-hardened age-hardened	С	260 °C (500 °F)	822 753	119.2 109.2	100	42	no no
age-hardened age-hardened	D	232 °C (450 °F)	822 753	119.2 109.2	100	61	no no
age-hardened age-hardened	E	RT	822 753	119.2 109.2	100	42	no no

\*Samples were coupled to carbon steel (screw)

Test Media:

5% NaCl + 0.23% acetic acid + 0.4% sodium acetate; Gasphase: saturated H<sub>2</sub>S, purging N<sub>2</sub>, atmospheric pressure А

5% NaCl + 0.5% acetic acid; Gasphase: saturated H<sub>2</sub>S, purging N<sub>2</sub>, atmospheric pressure В

C 15 % NaCl; Gasphase: 0.7 bar H<sub>2</sub>S + 34.5 bar CO<sub>2</sub>, purging N<sub>2</sub>, overall pressure 70 bar; at RT: 70 bar; at 260 °C: 200 bar

 $\begin{array}{l} \text{D} \quad 15\% \text{ NaCl; Gasphase: 0.7 but } H_2\text{S} + 52 \text{ bar CO}_2, \text{ purging } \\ \text{D} \quad 15\% \text{ NaCl; Gasphase: 12 bar } H_2\text{S} + 52 \text{ bar CO}_2, \text{ purging } N_2 \\ \text{E} \quad 5\% \text{ acetic acid; Gasphase: } H_2\text{S saturated; purging } N_2 \\ \end{array}$ 

Table 7 – C-Ring Stress-Corrosion-Cracking tests according to NACE Standard TM 0177, Method C of solution-annealed and age-hardened Nicrofer 4320 Ti in various sour environments.

#### Applications

Nicrofer 4320 Ti finds application in oil and natural gas exploration and production particularly in "sour" ( $H_2S$  containing) environments.

Typical applications are:

- tool joints, completion tools, hangers and packers
- down-hole and surface gas-well components
- pump shafting and similar high-strength hardware particularly in marine environments and others containing both chlorides and sulfides.

#### Melt practice

Nicrofer 4320 Ti is melted in induction furnaces. The cast ingots are subsequently remelted using the electro-slag remelt (ESR) process before proceeding with hot working.

#### Fabrication and heat treatment

Nicrofer 4320 Ti can readily be hot- and cold worked and machined.

#### Heating

Workpieces must be clean and free from all kinds of contaminants before and during any heat treatment.

Nicrofer 4320 Ti may become embrittled if heated in the presence of contaminants such as sulphur, phosphorus, lead and other low-melting-point metals. Sources of such contaminants include marking and temperature-indicating paints and crayons, lubricating grease, fluids and fuels.

Fuels must be as low in sulphur as possible. Natural gas should contain less than 0.1 wt.-% sulphur. Fuel oils with a sulphur content not exceeding 0.5 wt.-% are suitable.

Due to their close control of temperature and freedom from contamination, thermal treatments in electric furnaces under vacuum or an inert gas atmosphere are to be preferred. Treatments in an air atmosphere and alternatively in gas-fired furnaces are acceptable though, if contaminants are at low levels so that a neutral or slightly oxidizing furnace atmosphere is attained. A furnace atmosphere fluctuating between oxidizing and reducing must be avoided as well as direct flame impingement on the metal.

#### Hot working

Nicrofer 4320 Ti may be hot worked (forged) in the temperature range 1050 to 920 °C (1920 to 1680 °F), followed by air cooling.

Hot rolling should be carried out at temperatures  $\ge$  950 °C ( $\ge$  1740 °F).

For heating up, workpieces may be charged into the furnace at maximum working temperature. When the furnace has returned to temperature, the workpieces should be soaked for 60 minutes per 100 mm (4 in.) of thickness. At the end of this period it should be withdraw immediately and worked within the above temperature range. If the metal temperature falls below the minimum hot working temperature, it must be reheated.

#### Cold working

Cold forming of Nicrofer 4320 Ti is preferably done in the solution-annealed condition prior to age-hardening. Nicrofer 4320 Ti has a higher work-hardening rate than austenitic stainless steels. This should be taken into account when selecting forming equipment.

#### Heat treatment

Solution heat treatment should be carried out in the temperature range 980 – 1040 °C (1800 – 1900 °F) for 2 h followed by water quenching. For age hardening the following two step precipitation treatment process is used: 740 °C (1365 °F) for 6 h followed by furnace cooling to 621 °C (1150 °F) at which temperature the material is held for a further 6 h before air cooling to room temperature. This thermal treatment results in a structure with a homogeneous and uniformly distributed grain size.

For any thermal treatment the material should be charged into the furnace at maximum working temperature. Also for any thermal treatment operation the precautions concerning cleanliness mentioned earlier under 'Heating' must be observed.

#### Descaling and pickling

Oxides of Nicrofer 4320 Ti and discoloration adjacent to welds are more adherent than on stainless steels. Grinding with very fine abrasive belts or discs is recommended. Care should be taken to prevent tarnishing.

Before pickling which may be performed in a nitric/hydroflouric acid mixture with proper control of pickling time and temperature, the surface oxide layer must be broken up by abrasive blasting or by carefully performed grinding or by pretreatment in a fused salt bath.

#### Machining

Nicrofer 4320 Ti can be machined in the solution-annealed or age-hardened condition. As the alloy exhibits a high workhardening rate only low cutting speeds should be used compared with low-alloyed standard austenitic stainless steels. Tools should be engaged at all times. An adequate depth of cut is important in order to cut below the previously formed work-hardened zone.

Best results with the smoothest surface finish at final dimension are obtained by rough machining before age hardening and finishing after precipitation treatment in the aged condition. Nicrofer 4320 Ti is more easily machined than Nicrofer 5219 Nb – alloy 718 (UNS N07718).

#### Welding

Consultation with ThyssenKrupp VDM's Welding Laboratory is recommended.

#### Availability

Nicrofer 4320 Ti is available in the following standard product forms:

#### Rod & bar

#### Conditions:

forged, rolled, drawn,

thermally treated (solution annealed and age-hardened), pickled, machined, peeled or ground

Product	Forged <sup>1)</sup> mm	Rolled <sup>1)</sup> mm	Drawn <sup>1)</sup> mm					
Rod (o. d.)	≤ 250	8-60	12 – 50					
Bar, square (a)	40 – 175	15 – 175	not standard					
	inches	inches	inches					
Rod (o. d.)	≤ 10	$5/_{16} - 2^3/_8$	<sup>1</sup> / <sub>2</sub> - 2					
Bar, square (a)	1 <sup>5</sup> / <sub>8</sub> - 7	$^{10}/_{16} - 7$	not standard					
<sup>1)</sup> other sizes and cond	<sup>1)</sup> other sizes and conditions subject to special enquiry							

#### Forgings

Shapes other than discs, rings, rod and bar are subject to special enquiry. Flanges and hollow shafts may be available up to a piece weight of 4.5 t.

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