# Propustna in lezna krhkost CrMoV jekla z okoli 1 % Cr zaradi oligoelementov

Temper and crep embrittlement of a CrMoV steel with about 1 % chromium by residual elements

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Eksperimentalno delo smo izvršili z jeklom 28 Cr-Mo-Ni-V 4 9 (Stahl - Eisen - Werkstoffblatt 555), ki ima približno kemično sestavo: 0,28 % C, 1 % Cr, 0,9 % Mo, 0,7 % Ni in 0,3 % V. To je eno izmed dveh najpomembnejših jekel, odpornih proti lezenju, ki ju uporabljamo za rotorje srednje in visokotlačnih parnih turbin. Cilj raziskave je bil ugotoviti stopnjo popustne krhkosti, ki jo povzroče različne količine fosforja in kositra. Vzorci jekla so bili toplotno obdelani na martenzitno in bainitno strukturo. Nadalje smo ugotovili vpliv bakra in aluminija na prelomno duktilnost po lezenju in po različnih časih na temperaturah, pri katerih to jeklo uporabljamo.

Na osnovi več raziskovalnih del je znano, da je reverzibilna popustna krhkost odvisna od stopnje izcejanja elementov P, Sn, As in Sb po kristalnih mejah<sup>1</sup>. Ti elementi znižujejo energijo kristalnih mej<sup>2, 3</sup> in s tem tudi energijo interkristalnega preloma<sup>4, 5</sup>. Ugotavljajo, da povečujejo krhkost elementi v naslednjih redih P-Sn-Sb-As<sup>6</sup> ali Sb-P-Sn-As<sup>7</sup>. Z Augerjevo spektroskopijo so bile ugotovljene izceje elementov P in Sn na kristalnih mejah krhkih jekel<sup>8</sup>. Nadalje je znano, da popustno krhkost povečajo Ni, Mn in Si, verjetno zato, ker ti elementi premaknejo ravnotežje med Mo v raztopini in Mo v karbidih na stran Mo karbidov<sup>8</sup>.

Pomemben je zlasti Mo, ki preprečuje vpliv P. Zmanjšanje koncentracije Mo v raztopini povečuje izceje P na kristalnih mejah in s tem tudi krhkost. Razen Mo vplivata enako tudi Cr in V, vendar manj močno kot Mo<sup>9</sup>.

Ker tvori ogljik karbide s Cr, Mo in V, je bilo ugotovljeno, da je vsaka važnejša komponenta kemične sestave v tem jeklu direktno ali indirektno povezana z reverzibilno popustno krhkostjo.

Količin Cr, Ni in Mn ne moremo preveč znižati, ker so potrebni za kaljivost, da se izognemo nastajanju ferita v jeklu velikih odkovkov. Mo in nekoliko manj tudi V povečata kaljivost in odpornost proti lezenju, Ugotavljajo, da je potrebna količina teh elementov v raztopini 0,2 do 0,25 % tudi še po dolgih časih obratovanja pri delovni obremenitvi in temperaturi, ne glede na njuno vsebnost v talini<sup>10</sup>. Ni še gotovo, da je ta količina obeh elementov zadostna za preprečitev popustne krhkosti. Z ozirom na ozke meje elementov C, Ni, Experimental work has been carried out on the steel 28 Cr-Mo-Ni-V 4 9 according to the German Stahl-Eisen-Werkstoffblatt 555 with a nominal chemical composition of about 0.28 % C, 1 % Cr, 0.9 % Mo, 0.7 % Ni and 0.3 % V, beeing one of the two most important creep resisting forging steels used for intermediate and high pressure steam turbine rotors. The aim of the investigations was to find out the degree of temper embrittlement of this steel caused by certain amounts of phosphorus and tin, in a martensitic and in a bainitic completely heat treated structure. Further, an influence was to be confirmed of copper and aluminium on the rupture ductility after creep of some duration under service temperature.

It is known from numerous research works that reversible temper embrittlement corresponds with the degree of segregation of the trace elements P, Sn, As and Sb along the grain boundaries1. These elements are known to lower grain boundary energy 2, 3 and subsequently the intergranular fracture energy4,5. The embrittling potency is reported to increase in the order P-Sn-Sb-As6 or Sb-P-Sn-As7. Segregation of the elements P and Sn has been detected at the grain boundaries of embrittled steel by Auger-spectroscopy8. Further, it is known that temper embrittlement is enchanced by Ni, Mn and Si probably because these elements shift the equilibrium between molybdenum in solution and molybdenum in carbides to the side of molybdenum carbides8.

Molybdenum is of importance because it is a scavenger for phosphorus and therefore a reduced concentration of molybdenum in solution leads to a higher phosphorus segregation at the grain boundaries resulting in a higher trend to embrittlement. As compared to molybdenum the interaction of chromium and vanadium with phosphorus is not so strong<sup>9</sup>.

Because of the role of carbon in forming carbides with chromium, molibdenum and vanadium, it can be stated, that any of the important components of the chemical composition of the steel under consideration is directly or indirectly connected with the reversible temper embrittlement.

From these components, Cr, Ni and Mn should not be reduced too much, because they are necessary to give a sufficient hardenability so that ferrite in the core of big forgings is avoided. Molybdenum raises the hardenability and, next to vanadium, the creep resistance. It has been repor-

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Mn, Cr, Mo in V v teh jeklih nastaja vprašanje, katere količine drugih elementov lahko dopuščamo brez nevarnosti za popustno krhkost. V našem delu smo se osredotočili na Si, P in Sn.

V 45-kilogramski laboratorijski indukcijski peči smo izdelali taline, katerih osnovna sestava je navedena v tabeli 1. Številke kažejo najvišjo in najnižjo vsebnost legirnih elementov osmih talin, ki smo jih rabili pri prvih preizkusih našega programa.

# Tabela 1: Kemična sestava preizkušanih jekel. Osnova: 28 Cr-Mo-Ni-V 4 9 (%)

Table 1: Chemical composition of experimental steels. Base: 28 Cr-Mo-Ni-V 4 9 (%)

Os	novna	sestava 8			reizkušanih j	ekel		
Base	comp	osition	of	8	experimental	steels		

С	Mn	S	Cr	Mo	Ni	v	As	Sb	N
0,29	0,56	0,007	0,94	0,90	0,64	0,31	0,004	0,001	0,008

0,33 0,70 0,009 1,03 0,95 0,68 0,35 0,005 0,002 0,010

Dodatno:

Additionally:

Talina Melt	Si	Р	Sn	Al	Cu	
1	0,06	0,006	0,005	0,003	0,04	
3	0,05	0,005	0,005	0,003	0,16	
5	0,06	0,005	0,013	0,025	0,04	
7	0,04	0,005	0,005	0,044	0,03	
14	0,05	0,024	0,017	0,003	0,03	
15	0,53	0,025	0,017	0,003	0,04	
19	0,63	0,074	0,018	0,017	0,03	
20	0,64	0,082	0,017	0,003	0,04	

Ingoti so bili skovani v palice, ki so bile toplotno obdelane za prvo serijo preizkusov na martenzitno strukturo, za drugo serijo pa na bainitno strukturo. Preizkusne palice z martenzitno strukturo so bile po popuščanju hlajene v olju za pridobitev začetnega žilavega stanja materiala kot osnove za krhkostne preizkuse. Preizkusi z bainitno strukturo pa naj bi simulirali stanje materiala pri velikih turbinskih rotorjih. Toplotna obdelava je bila torej izbrana v skladu s časovno-temperaturno krivuljo za turbinski rotor, premera 1000 milimetrov, če je ta kaljen z 970 °C v olju in popuščen. S stopenjskim hlajenjem pa smo preizkusili, koliko ta vpliva na pomik FATT (Fracture Appearance Transition Temperature - prehodna temperatura žilavosti, določena po videzih prelomnih površin).

Tabela 2 vsebuje rezultate vzorcev z martenzitno strukturo. Niti baker (talina 3) niti kositer (talina 5) v količinah, ki jih lahko najdemo v odted to remain in solution at a level of 0.2 to 0.25 % irrespective of the initial bulk content of the melt, after long time exposure at service temperature and load<sup>10</sup>. It is not certain, whether this level is sufficient to avoid temper embrittlement in 1 % Cr-Mo-V steel. On the basis of the assumption that the chemical composition of melts intended for big creep resistant forgings should remain within narrow limits regarding the elements C, Ni, Mn, Cr, Mo und V, the question arises, what level of other elements can be tolerated without running a risc of temper embrittlement. In the first step, we concentrated on Si, P, and Sn.

45 kg laboratory induction furnace melts have been made with a base composition shown in table 1. The figures in the upper line give the highest and lowest content of the alloying elements of the eight melts used in the first step of our programme.

The ingots have been forged to bars and heat treated to obtain a martensitic microstructure for one series of the tests and a bainitic microstructure for another series. The martensitic test bars have been oil cooled after tempering to give a tough initial state of the material as a basis for embrittling experiments. The tests with bainite have been done to simulate the state of material near the rim of a big turbine rotor. Therefore, a heat treatment was choosen similar to the timetemperature curve near the rim of a turbine rotor of 1000 mm diameter when austenitized at 970 °C, quenched in oil and tempered production-like. Step cooling has been used to developed a shift in FATT if possible (FATT - Fracture Appearance Transition Temperature).

Table 2 represents the results obtained on martensitic specimens. Neither copper (melt 3) nor tin (melt 5) in amounts possibly encountered in forgings, change the FATT, and no shift of FATT was produced by step cooling. If a relatively high phosphorus — and tin — content are combined (melt 14), there may be some indication of an increase of FATT by step cooling. If the silicon content is increased (melt 15), an embrittlement becomes more distinct. At high silicon — and phosphorus — contents i. e. beyond technical limits (melt 19 and 20), an embrittlement by step cooling ist quite obvious.

Table 3 represents the results obtained on bainitic specimens showing that silicon and phosphorus contents beyond the technical level and a relatively high tin content result in temper embrittlement in bainitic structure of a turbine rotor. It should be pointed out, that the structure tested here consisted of a mixture of lower and upper bainite with lower bainite being predominant. Moreover, it might be concluded that no temper embrittlement — as revealed by step cooling — would develop in a fully upper bainitic microstructure like that near the core area of a kovkih, ne spremenita FATT. Tudi ni opaziti nobene spremembe FATT pri stopenjskem ohlajanju. V primeru sorazmerno visokih vsebnosti P in Sn (talina 14) je nekaj indikacij, da se pri stopenjskem ohlajanju poviša FATT. Če se poveča količina Si (talina 15), postane krhkost bolj očitna. Pri količinah S in P nad tehničnimi mejami (talini 19 in 20) postane krhkost po stopenjskem ohlajanju zelo izrazita.

## Tabela 2: Vpliv Si, P in Sn na zarezno žilavost. Jeklo 28 Cr-Mo-Ni-V 4 9

Table 2: Influence of Si, P and Sn on notch toughness. Steel 28 Cr-Mo-Ni-V 49

Talina Melt	Vsebnost oligoelementov Content of residual elements %	R <sub>m</sub> 20 ℃ N/mm²	FATT °C olje Oil	∆FATT <sup>®</sup> C Stopenj- sko hlajeno Step cooling	
1		760	93	0	
3	0,16 Cu	785	98	0	
5	0,013 Sn	790	95	$\sim 0$	
7		790	80	0	
14	0,024 P 0,017 Sn	770	90	30	
15	0,53 Si 0,025 P 0,017 Sn	790	-72	50	
19	0,63 Si 0,074 P 0,018 Sn	810	-40	135	
20	0,64 Si 0,082 P 0,017 Sn	810	55	135	

Toplotna obdelava, heat treatment: 970° 3 ure, hrs/olje, oil, + 720° 15 ur, hrs/olje, oil, presek, section  $25 \times 25$  mm

Mikrostruktura, microstructure: Martenzit, martensite

Iz tabele 3, ki vsebuje rezultate vzorcev z bainitno strukturo, je razvidno, da je potrebno več Si in P, kot ju vsebujejo tehnična jekla, in sorazmerno visoka vsebnost Sn, da se pojavi popustna krhkost pri bainitni strukturi turbinskega rotorja. Treba je poudariti, da je bila struktura, ki jo obravnavamo, mešanica spodnjega in zgornjega bainita, z večino spodnjega. Celo lahko zaključimo, da se popustna krhkost ne bo razvila (kot se kaže pri stopenjskem ohlajanju) v popolnoma zgornji bainitni strukturi, kot je tista v jedru odkovka, če je kemična sestava ista, kot smo jo že omenili.

Prvi primer krhkega loma zaradi lezenja je znan s konca tridesetih let, ko so se na prvem navoju pri glavi lomili prirobniški vijaki pri visokotlačnih parnih ceveh pri delovni temperaturi 500 °C. Vijaki so bili izdelani iz jekla s približno 0,12 % C, 1,5 % Ni, 0,7 % Cr in 0,5 % Mo in so bili poboljšani na natezno trdnost okoli 900 N/mm<sup>2</sup>. Sprva so verjeli, da sta za te lome odločilni vsebnost niklja in neke vrste popustna krhkost. Danes big forging, provided that the chemical composition lie within the limits mentioned above.

Tabela 3: V pliv Si, P in Sn na zarezno žilavost. Jeklo 28 Cr-Mo-Ni-V 4 9

Table 3: Influence of Si, P and Sn on notch toughness. Steel 28 Cr-Mo-Ni-V 49

Talina Melt	Vsebnost oligoelementov Content of residual elements %	R <sub>ss</sub> 20 ℃ N/mm²	FATT °C	ΔFATT °C Stopenj- sko hlajeno Step cooling
1		770	+ 75	0
3	0,16 Cu	795	+ 62	~ 0
5	0,013 Sn	815	+ 65	~ 0
7		805	+ 76	+10
14	0,024 P 0,017 Sn	770	+ 80	~ 0
15	0,53 Si 0,025 P 0,017 Sn	825	+ 92	+25
19	0,63 Si 0,074 P 0,018 Sn	820	+104	+60
20	0,64 Si 0,082 P 0,017 Sn	830	+110	+60
Top hrs/	lotna obdelava, heat rob, border 1000 mm,	treatme hlajeno	nt: 970 v olju,	<sup>0</sup> , 18 ur, oil cool-

ing, 710° 10 ur, hrs/35° na uro, pro hr Mikrostruktura, microstructure: bainit, bainite

The first cases of brittle creep failure became known at the end of the thirties when high pressure steam tube flange bolts at a service temperature of 500 °C fractured in the first thread of the screw head. The bolts were made of a steel with about 0.12 % C, 1.5 % Ni, 0.7 % Cr and 0.5 % Mo,



Lezna lomna trdnost pri duktilnem in zarezno občutljivem stanju jekla (shematično)



Creep rupture strength at ductile and notch sensitive steel state (shematically)

je znano, da taki lomi lahko nastanejo pri vrsti kovinskih materialov, od nizko legiranih konstrukcijskih jekel do jekel, odpornih proti lezenju, zlitin na osnovi niklja in do neželeznih zlitin. Slika 1 daje osnovno razlago takega loma. V zgornjem delu slike so prikazani rezultati preizkusov lezne lomne trdnosti za žilavo stanje materiala: pri vseh časih in enakih obremenitvah je pri zarezanih vzorcih čas loma daljši kot pri gladkih vzorcih. Stevilke pri gladkih vzorcih so lomni raztezki. Vrednosti so zadosti visoke in se ne spreminjajo sistematično. Na spodnjem delu slike pa so prikazani rezultati preizkusov lezne lomne trdnosti za krhko stanje materiala. Opazimo lahko dva značilna pojava: v določenem intervalu, katerega trajanje je odvisno od stanja materiala in razmer prezkušanja lezenja, je pri zarezanih vzorcih čas do loma pri enakih obremenitvah krajši kot pri gladkih vzorcih in lomni raztezki gladkih vzorcev





Vpliv natezne trdnosti, bakra in aluminija na lezno zarezno občutljivost pri Cr-Mo-V jeklih z okoli 1% Cr



Influence of tensile strength, cooper and aluminium on creep limit notch sensitivity of Cr-Mo-V steels with appr. 1% Cr heat treated to a tensile strength of about 900 N/mm2. At first, the nickel content and some kind of temper embrittlement were believed to be responsible for the failures. Today, however, it is known, that failures of this kind may be observed at a broad variety of metallic materials, ranging from low alloy construction steels to creep resistant steels, nickel base alloys and to non ferrous alloys. Figure 1 shows the principal features of this failure. In the upper part of the picture results of creep rupture tests at a tough state of material are shown: at any time, the rupture lifes of notched specimens are longer than those of plain specimens at the same load. The figures at the plain specimens represent the rupture elongations. The values are sufficiently high and do not change in a systematic manner. In the lower part of the picture, results of creep ruptur tests at a brittle state of the same material are represented. Two typical features may be observed: At a certain interval, the duration of which depends on the state of the material and the test conditions, the rupture times of notched specimens are shorter than those of plain specimens with the same load, and the rupture elongations of the plain specimens, in the same interval, decrease to very low values. The underlying mechanism includes the relative creep strength of grain volume and grain boundary and further aspect which will be mentioned later on. In addition to that, sometimes an influence of impurities has been presumed or found in experiments. Since 1967, scarce references have been made regarding an influence of aluminium or copper on the rupture ductility: Ratcliff and Brown11 reported about a deleterious effect of 0.020 % soluble aluminium on the rupture ductility of a 1 % Cr-Mo-V steel. Their specimens had tensile strengths of about 1100 to 1300 N/mm2 which is outside technical limits. Beneš and Skvor12 noted a decrease in rupture ductility of a Cr-Mo-V Steel in creep tests at 600 °C by 0.25 % Cu. Hopkins and al13 investigated a steel similar to 28 Cr-Mo-Ni-V 4 9 with bainitic microstructure and found longer rupture life and higher rupture ductility for a high purity laboratory bar material as compared to steel of conventional purity. The specimens had tensile strengths of about 1000 N/mm2 which is too high for this type of steel. As a consequence, the rupture elongations of the bainitic specimens remained low for the pure steel (6 % at 13 000 hrs rupture time) as for the impure steel as well (3.9 % at 4500 hrs rupture time). Viswanathan, in his review article14, again mentioned the influence of Al and Cu, among others on rupture ductility.

We had the opportunity to use a great amount of data kindly submitted by the German Cooperative Creep Testing Committee to find out whether a relation exists between the copper and aluminium content of ruptured creep specimens and their rupture behaviour. A qualitative relationship could be found. Figure 2 shows a diagram based

se v istem intervalu znižajo do zelo majhnih vrednosti. Osnovni mehanizem vsebuje relativno trdnost lezenja notranjosti zrna in kristalne meje. nadaljnji vidiki pa bodo omenjeni kasneje. Dodatno k temu pa včasih domnevajo tudi na vpliv nečistoč, ali pa so ta vpliv ugotovili v eksperimentih. Od leta 1967 je le malo podatkov o vplivu aluminija ali bakra na lomno duktilnost. Ratcliff in Brown11 poročata o škodljivem vplivu 0,020 % topnega Al na lomno duktilnost 1 % Cr-Mo-V jekla. Njuni vzorci so imeli natezno trdnost 1100 do 1300 N/mm2, kar je zunaj tehničnih mej. Beneš in Skvor12 poročata, da 0,25 % Cu v Cr-Mo-V jeklu zniža lomno duktilnost pri preizkusih lezenja na temperaturi 600 °C. Hopkins in sodelavci13 so ugotovili pri jeklu, podobnem 28 Cr-Mo-Ni-V 4 9, ki je imelo bainitno strukturo, daljši čas do loma in večjo lomno duktilnost, če je bilo to jeklo izdelano laboratorijsko in zelo čisto, v primerjavi z jeklom običajne čistoče. Vzorci so imeli natezno trdnost okoli 1000 N/mm2, kar je preveč za to vrsto jekla. Posledica je bil nizek lomni raztezek pri čistem jeklu (6 % po 13000 urah do loma) in prav tako tudi pri nečistem jeklu (3,9 % po 4500 urah do loma). Tudi Viswanathan v svojem delu14 omenja vpliv Al in Cu ter drugih elementov na lomno duktilnost.

Priložnost imamo uporabiti veliko podatkov, ki jih je prijazno dal na razpolago nemški Komite za preizkušanje lezenja, iz katerih je razvidna zveza med vsebnostjo Cu in Al v vzorcih in njihovim vedenjem pri lomih. Ta najdena zveza je kvalitativna. Slika 2 kaže diagram, osnovan na rezultatih okoli 120 serij preizkusov lezenja različnih Cr-Mo-V jekel. Ordinata predstavlja natezne trdnosti vzorcev, x-os pa poljubno kombinacijo vsebnosti Cu in Al. Temperatura preizkušanja lezenja je bila večinoma 550 °C. Če so bili raztezki ali kontrakcije po lomu majhni, ali če so bili časi do loma zarezanih vzorcev krajši od tistih pri gladkih vzorcih (pri isti obremenitvi), je rezultat preizkusa označen s trikotno točko. Žilavi lomi so označeni s krogi. Vidi se, da mora biti pri večjih vsebnostih Cu in Al natezna trdnost nižja, da dobimo žilav prelom, in obratno. V to shemo je zajetih 80 % preizkusnih serij.

Predpostavljamo, da Al in Cu ne znižujeta površinske energije faz na kristalnih mejah, kot so karbidi ali druge. Nastane vprašanje, na katerem mehanizmu je osnovana ta zveza med obema elementoma, natezno trdnostjo in prelomom vzorcev pri preizkusih lezenja. Da bi se temu posvetili bolj podrobno, smo napravili preizkuse lezenja s talinami različnih čistosti.

V tabeli 4 je preliminaren pregled dela teh preizkusov. Vključena je talina z veliko čistostjo in druga, ki vsebuje Cu in Al. V tabeli so navedena avstenitizacijska temperatura T<sub>A</sub>, natezna trdnost R<sub>m</sub> in rezultati preizkusov lezenja pri štirih nape-

on the results of about 120 series of creep tests of different Cr-Mo-V steels. The ordinate represents the tensile strength of the specimens, the x-axis represents some arbitrary combination of their copper and aluminium contents. The creep testing temperature was mainly 550 °C. If rupture elongations or reductions of area were of a few percents only or if the rupture life of a notched specimen was shorter than that of a smooth specimen at the same load, the result is marked by a triangle. Tough ruptures are marked by a circle. It can be seen that the higher the copper and aluminium content, the lower must the tensile strength be to give tough rupture and vice versa. 80 percent of the test series fit into the scheme, so there should be something significant behind it.

It is supposed, that aluminium and copper are not effective in reducing the surface energy of phases like carbides or others in the grain boundary. So the question arises whether there is some mechanism by which the relation between these elements, the tensile strength and the rupture behaviour of creep specimens is established. To follow this into more detail, we have carried out creep tests with melts of different purity. In table 4, a preliminary survey of a part of this investigation is given. A melt of high purity and another one with copper and aluminium impurities are included. The austenitizing temperature, TA, the tensile strength, Rm, and the results of creep tests at four different levels of creep load are noted. The creep temperature was 550 °C in all cases. Combined specimens with a plain and a notched part were used. The rupture life of both parts is given. Besides, the rupture elongation (A) and reduction of area (Z) are also given.

970 °C is generally accepted as being the upper limit of austenitizing temperatures for this steel in practice. It is seen, that, using it, a tensile strength of 1140 N/mm2 is too high, if creep embrittlement should be avoided even in a high purity metal 1050 °C is known to be an austenitizing temperature enchancing creep embrittlement, if the material is tempered too low and hence has a high tensile strength. From line 2 and 3 it may be deduced, that under these circumstances, a tensile strength of 970 N/mm2 is too high, and leads to creep embrittlement while this seems not to be the case with a tensile strength of 770 N/mm2. With the impure material, an austenitizing temperature of 1050 °C, which is known to be too high, leads to embrittlement rather early even at a tensile strength of 830 N/mm<sup>2</sup>, which is within the practical limits. A lower austenitizing temperature of 890 °C has to be used and rather impractical low tensile strengths, namely 550 or 650 N/mm2, to avoid creep embrittlement.

Temper embrittlement and creep embrittlement follow different mechanisms. As to the first, the cohesive forces between adjacent grains are lowered by impurities such as P, Sn, Sb, and others, which segregated to the grain boundaries.

		P		Napet	ost, St	tress, N/n	nm²			
	TA	N/mm <sup>2</sup>	400	310		260		200		150
			0 366		0	3555	0	6020		
			A 565		Δ	3380	Δ	5571		
	970°	1140	A 14.7		Α	6,9	A	5,5		
0,01 Cu			Z 29,5		Z	8,5	Z	3,7		
< 0.003 A1					0	838	0	1788		
					Δ	208	Δ	486	1	0
0.001 Sb		970			A	1.5	A	1.2	-	$\rightarrow 100$
0,005 Sn					Z	3,5	Z	0,6	9	Δ
	1050° -			0 176	0	744	-			
				A 412	Δ	989			(	0
		770	_	A 28.5	A	15.9			7	$\rightarrow 100$
				Z 76,6	Z	44,2				Δ
					0	32	0	149		
					Δ	292	Δ	864		
		550			Α	38,1	A	43,4		_
0,15 Cu	0000				Z	80,4	Z	79,8		
0,031 AI 0,003 Sb	890" -						0	839	0	7386
0.014 Sn							Δ	2969	$\Delta$	7698
		650	_				A	21.1	A	13,6
							Z	63,1	Z	31,6
				0 208	0	931	0	6217		100:
				△ 299	Δ	514	Δ	5426	0	29694
	10500	830	_	A 15,5	A	6,4	A	6,6	Δ	19012
				Z 35,6	Z	3,9	Z	15,6	A	9,1
									Z	11,4

Tabela 4: Preizkusi lezenja za pojav lezne krhkosti Table 4: Creep experiments for the process of creep embrittlement

Gladki vzorci, plain specimens

△ Zarezani vzorci, notched specimens

tostih. Temperatura preizkušanja je bila v vseh primerih 550 °C. Uporabljeni so bili gladki in zarezani vzorci. Pri obeh vrstah vzorcev so navedeni časi do preloma. Razen tega so v tabeli zabeleženi tudi raztezki (A) in kontrakcije (Z).

V splošnem velja, da je za to jeklo v praksi zgornja temperatura avstenitizacije 970 °C. Če uporabimo to temperaturo, kaže, da je natezna trdnost 1140 N/mm<sup>2</sup> previsoka, če se hočemo celo pri zelo čisti talini izogniti krhkosti pri lezenju. Znano je, da temperatura avstenitizacije 1050° C poveča krhkost pri lezenju, če je bil material prenizko popuščen in ima zato visoko natezno trdnost Iz druge in tretje vodoravne vrste v tabeli 4 lahko sklepamo, da je v teh okoliščinah natezna trdnost 970 N/mm<sup>2</sup> previsoka in vodi do lezne krhkosti, medtem ko do tega ne pride pri natezni trdnosti 770N/mm<sup>2</sup>. Pri nečistem materialu povzroči avstenitizacijska temperatura 1050 °C krhkost celo pri natezni trdnosti 830 N/mm<sup>2</sup>, ki je znotraj prakAs to the second, cavity nucleation at grain boundary carbides or other phases during grain boundary creep plays the main role. On the other hand, the cohesive force between grain boundary phases and the matrix will also be lowered by impurities. It is known that brittle intergranular fracture has its origin in nucleation and growth of cavities mainly at the interface between carbides and matrix. Now the critical free energy  $\Delta G$  of cavity nucleation is

$$\Delta G = \frac{4\gamma^3 F}{\sigma^2},$$

were  $\gamma$  is the surface energy per unit area, F a geometrical factor, and  $\sigma$  the stress acting at the place of the nucleating cavity. Due to the exponent 3 of  $\gamma$ ,  $\Delta G$  is highly sensitive to changes of the surface energy, for instance by impurities. Moreover, the matrix with a higher strength has the higher average stress along the grain boundary and subsequently higher void growth rates<sup>16</sup>. This tičnih mej. Treba je bilo uporabiti nižjo avstenitizacijsko temperaturo 890 °C in jeklo popustiti na prenizke natezne trdnosti 550 ali 650 N/mm<sup>2</sup>, da se ni pojavila lezna krhkost.

Popustna in lezna krhkost sledita različnim mehanizmom. Prvič, elementi P, Sn, Sb in drugi, ki se izcejajo na kristalnih mejah, znižajo kohezivne sile med sosednjimi zrni. Drugič, med lezenjem po kristalnih mejah igra najvažnejšo vlogo nukleacija praznin pri karbidih in drugih fazah na kristalnih mejah. Na drugi strani pa nečistoče znižajo kohezivne sile med fazami na kristalnih mejah in matrico. Znano je, da ima krhki interkristalni prelom izvor pretežno v nukleaciji in rasti praznin na mejni ploskvi med karbidi in matrico. Kritična prosta energija  $\Delta G$  za nukleacijo praznin je

$$\Delta G = \frac{4\gamma^3 F}{\sigma^2},$$

kjer je  $\gamma$  površina energije na enoto ploskve, F geometrijski faktor in  $\sigma$  napetost, ki deluje na mestu nukleacije praznine. Zaradi eksponenta 3 pri  $\gamma$  je  $\Delta G$  zelo občutljiv za spremembe površinske energije, na primer zaradi nečistoč. Poleg tega ima trdnejša matrica večje povprečne napetosti vzdolž kristalnih mej in s tem večje hitrosti rasti praznin<sup>16</sup>. To je v skladu z opažanji, da je krhki lom pri lezenju bolj verjeten pri visoki trdnosti jekla kot pri nizki. Vlogo določenih nečistoč pri nastajanju lezne krhkosti moramo še nadalje zasledovati, zlasti s preizkusi lezenja pri materialih z martenzitno mikrostrukturo.

#### RAZPRAVA

### C. Goux, ENSM, Saint Etienne

Kakšen je razlog za nizko vsebnost silicija, ki je omenjena v eni od vaših slik?

## M. Krause

Nizko vsebnost silicija v nekaterih od preizkušenih materialov smo izbrali, da bi določili, če silicij pospešuje popustno krhkost v jeklih te vrste. Na drugi strani je mogoče, da bo to jeklo dezoksidirano po postopku vakuumske dezoksidacije z ogljikom na talinah, ki so praktično brez dodatka silicija.

## S. Engineer, Thyssen Edelstahlwerke, Krefeld

Kako pomembna je udarna žilavost pri sobni temperaturi za lezne lastnosti in prelom teh jekel?

## M. Krause

Če mislite kako značilne so nekatere žilavosti pri sobni temperaturi s stališča leznih lastnosti pri delovni temperaturi, naj vas spomnim, da ima veis in agreement with the observation that brittle creep failure is more likely to occur at high tensile strength than at a low one. Therefore, the effect of certain impurities on creep embrittlement shall be investigated mainly with creep tests on materials with martensitic microstructure which had shown temper embrittlement as mentioned above.

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#### DISCUSSION

## C. Goux, ENSM, Saint Etienne

What is the reason for a very low silicon content mentioned in one of your slides?

#### M. Krause

The low silicon content in some of the test materials was chosen as a basis for determining whether silicon might promote temper embrittlement in this type of steel. On the other hand, it could be possible that this steel will be deoxidised by the vacuum carbon deoxidation procedure which would be done on heats with nearly no addition of silicon.

#### S. Engineer, Thyssen Edelstahlwerke, Krefeld

How important are the impact toughness properties at room temperature for the creep rupture properties of these steels?

#### M. Krause

If you mean how indicative certain values of the room temperature impact toughness are regarding the creep rupture properties at service temperature, you may recollect, that high impact liko udarno žilavost mikrostruktura iz martenzita ali spodnjega bainita, ki pa imata oba majhno lezno trdnost, medtem ko ima mikrostruktura iz zgornjega bainita nizko žilavost in največjo odpornost proti lezenju v jeklih te vrste.

Zato so sprejemljive nizke žilavosti do 10 J za izkovke za turbinske rotorje za delo pri visoki temperaturi.

## F. Vodopivec

Imam vprašanje, ki morda ni neposredno povezano s temo vašega predavanja. V ohišjih ventilov za visokotlačne parne kotle smo opazili, da se relativno velike razpoke (dolžine nad 100 mm, globine do 15 mm) lahko razvijejo med letnimi ali dvoletnimi revizijami. Ohišja so iz jeklene litine podobne sestave, kot jekla katera obravnavate v vašem delu, le vsebnost ogljika je nižja, okoli 0,15 %. Delovna temperatura je približno 515 °C, pritisk pa 125 at. Elektrarna je namenjena za pokrivanje energetskih konic. Ali mislite, da so razpoke izključno posledica malociklične utrujenosti, ali je vmes tudi vpliv krhkosti?

## M. Krause

Ohišja ventilov so obremenjena z malociklično utrujenostjo, posebno v koničnih centralah. To bi bilo bolj nevarno za krhko kot za žilavo jeklo. Kaj je v Vašem primeru razlog za pokanje se lahko določi samo na osnovi mikrostrukturnih raziskav, popolnega poznavanja mehanskih lastnosti, kemične sestave, termične obdelave in delovnih karakteristik ventila. toughness is correlated with martensite or martensite plus lower bainite, both with poor creep resistance, and upper bainite, with low impact values, represents the microstructure with the highest creep resistance in this type of steel. Therefore, if 1 % Cr-Mo-V turbine rotor forgings for high temperature service are considered, impact values as low as about 10 J will be sufficient.

## F. Vodopivec

I have a question which is not maybe directly related to the subject of your paper. We observed that in valve casings for high pressure steam power station relatively great craoks (length over 100 mm and depth to 15 mm) could develop between annual or biannual revisions. The casings are manufactured from cast steel of similar composition as the steel which you discussed in your communication, only the content of carbon is lower, appr. 0.15 %. The working temperature is appr. 515 °C and the pressure 125 atm. The power station is intended to cover peak energy requirements. Do you mean that the cracks are due only to low cycle fatigue, or brittleness phenomena are involved also?

#### M. Krause

Valve bodies are exposed to low cycle fatigue especially in power stations covering consumption peaks. This would be more harmfull in a brittle material than in a tough one. But what, in your case, has been the reason for cracking can only be assessed by microstructural examinations and complete knowledge of the mechanical properties, the chemical composition, the heat treatment of the material and the service caracteristics of the valve.