EFFET DE LAMINAGE A CHAUD SUR LE COMPORTEMENT DE LA RECRYSTALLISATION DE L'ACIER INOXYDABLE FERRITIQUE TYPE 409.

EFFECT OF HOT ROLLING ON RECRYSTALLISATION BEHAVIOUR OF FERRITIC STAINLESS STEEL TYPE 409.

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RESUME: Le comportement de la recristallisation statique d'un acier inoxydable ferritique type 409 qui représente la phase ferritique à des températures élevées et qui contient des précipités a été examiné en utilisant des essais de laminage à chaud à une seule passe.

Les essais de laminage à chaud étaient réalisés à une température de laminage de 870°C jusqu'à une épaisseur finale de 8,4mm et 4,7mm suivi par un maintien à la même température de laminage.

Le volume recristallisé augmente avec l'augmentation de la réduction par laminage à chaud et le temps de maintien après laminage. La zone privilégiée pour cette recristallisation était les joints des grains précédents, en particulier les points triples des joints ou la déformation était bien concentrée. Ce matériau à taille de grain fine montre un rythme plus lent dans la recristallisation et ceci est attribué à la présence du titane.

Mots clés: Acier inoxydable ferritique type 409, précipités, variables de deformation restauration dynamique, restauration statique, recristallisation statique.

ABSTRACT : The behavior of the static recrystallization of this ferritic stainless steel type 409 which contains precipitates was examined using hot rolling test of a single pass. Hot rolling tests were carried out at temperature of 870°C from a thickness of 15mm to 8,4mm and 4,7mm thickness at a controled strain rate of 3.3s-1. The recrystallized volume increases with the increase of the reduction and the holding time after hot rolling. Nucleation of recrystallising grains appears to be associated with triple points and grain boundaries. This material displays a slower rate of recrystallisation and this is attributed to the presence of titanium.

Keywords: *ferritic stainless steel type 409, precipitates, deformation variables, hot rolling, dynamic recovery, static recovery, static recrystallization.*

1. INTRODUCTION

This research was carried out using ferritic stainless steel type 409 which was received in the form of hot rolled commercial plates with a uniform structure composed of equiaxed grains of size of 160μm after a complete recrystallisation. To obtain different starting grain size, slabs of this material were reheated at temperatures of 900^{o}C, 1100^{o}C and 1200^{o}C from 900

seconds up to 2400 seconds and immediately water quenched. The starting grain size prior to rolling operations was found to be independent of reheating temperature. A true activation energy for recrystallisation of 185 kJ.mol^{-1} was obtained.
In order to determine the isothermal recrystallisation kinetics after deformation to different effective strains at a strain rate of $3.3s^{-1}$ where dynamic recovery is the only restoration process during deformation, slabs of this material were deformed at temperature of 870^{o}C and effective strains of 0.58 and 1.16.
The recrystallisation rate which is characterized by the time required to achieve fifty percent transformation has been found for isothermal tests before the steady state is reached.
The power dependence of the recrystallised grain size on the strain applied was found.
Subgrain structure within deformed grains at a temperature of 870^{o}C is very poorly developed and not resolvable with the presence of undissolved coarse precipitates with different shapes.
Optical metallography of annealed specimens deformed at a temperature of 870^{o}C indicated that inhomogenieties of recrystallisation in the longitudinal sections occurred through the thickness and that the grain boundary regions were preferred sites for nucleation.

2. MATERIALS AND METHODS

Stainless steel type 409 was supplied in the form of hot rolled commercial plate, with thickness of 30mm and initial grain size of 160±10µm (Figure 1).

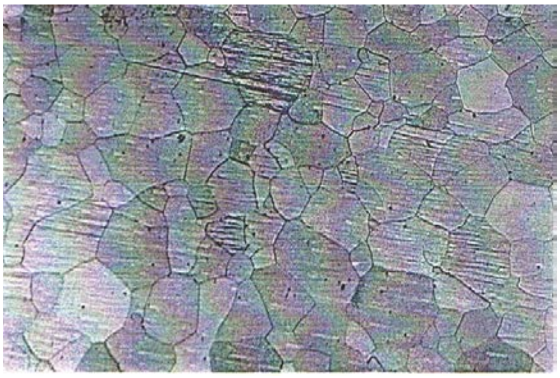

Figure 1. Optical micrograph of original structure of ferritic stainless steel type 409 supplied for the present work. X50

The commercial plate analysis is shown in table 1.

Element	Weight(%)
C	0,016
S	<0,001
P	0,014
Mo	0,03
Ni	0,2
Si	0,42
V	0,05
Cr	12,2
Mn	0,31
Nb	<0,02
Ti	0,32
Co	<0,02
Cu	0,04
N	161ppm

Table 1. Chemical composition of the ferritic stainless steel type 409 employed.

The plates were machined and ground to obtain specimens of dimensions of 120×30×15mm thick for the rolling experiments. Hot rolling experiments were performed with the laboratory 2-high, 50 tonnes rolling mill, measuring the temperature by a Pyrotenax Chromel-Alumel thermocouple to the centre of each slab so that the bead lay at the mid-length, mid-width and mid-thickness position. After reheating at temperature of 900°C for 20 minutes, the slabs were hot rolled at temperature of 870°C from a thickness of 15mm to 8.4mm and 4.7mm thickness at a controled strain rate of 3.3s^{-1}.

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The rolling schedules were given in a single pass sequence and then the slabs were water quenched after the pass to freeze the as-deformed structure, the time elapsed between the finishing pass and the quenching operation was of the order of 4 seconds. The time for which the specimens remained in the soaking furnace was recorded by stop watch. Because the slabs were quenched and then the specimens were heated over a finite time during subsequent annealing, a total equivalent time for recrystallisation was calculated by equation (1).

$$t_{eq}=\sum(\exp(-Q_{rex}/RT_i))dt/\exp(-Q_{rex}/RT) \quad (1)$$

teq is the equivalent time for annealing.
Ti is the temperature in a particular time interval dt.
T is the annealing temperature.
R is the universal gas constant, .
Qrex is the activation energy for static recrystallisation, which it is equal to 120 $kJ.mol^{-1}$ for stainless steel type 409.

After sectioning and linishing, specimens were ground down through a series of successively finer silicon carbide papers, 1200 grade was the finest used. Polishing was then performed on diamond wheels in the sequence 6, 1, 1/4µm grades. Various etches were tried electrolytically and chemically but most of these were unsatisfactory, since substructures in unrecrystallized grains were not revealed. The chemical etch of reagent containing 20ml HCl, 15ml H2o, 65ml CH3OH and 1g CuCl2 as well as reagent containing 50ml HCl, 5ml HNO3 and 50ml H2o was selected finally, since this reagent gave good grain boundary delineation and revealed substructures within the unrecrystallized grains, but it also produced some pitting using electro-etching. The specimens were also chemically etched with stirring the solution for a determined time until the grain boundary delineation was completely revealed. After taking the specimens out of the solution, they were rinsed with water followed by methyl alcohol, and dried in hot air.

3. RESULTS

In order to determine the isothermal recrystallisation kinetics after deformation to different effective strains at a strain rate of $3.3s^{-1}$ where dynamic recovery is the only restoration process during deformation, slabs of this material were deformed at temperature of 870°C and effective strain of 0.58 and 1.16.

The recrystallisation rate which is characterized by the time required to achieve fifty percent transformation $t_{50\%}$ is presented in figure 2. The power dependence of the relationship between $t_{50\%}$ and effective strain has been found for isothermal tests before the steady state is reached to correspond to the relation:

$$t_{50} \alpha \varepsilon^{-2.8} \quad (2)$$

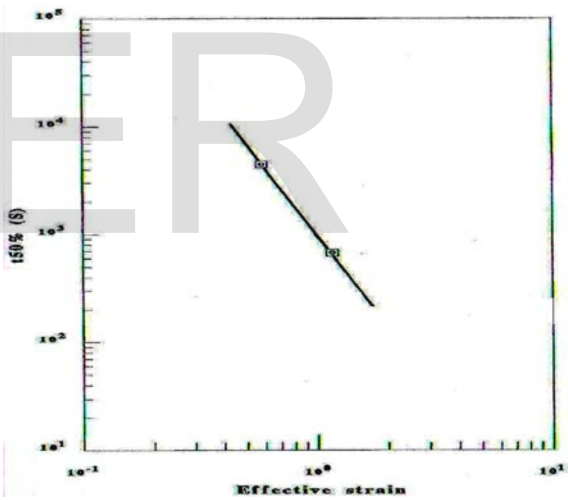

Figure 2. Effect of strain on time for 50% static recrystallisation of material of ferritic stainless steel type 409, deformed at a temperature of 870°C, d_0=160µm.

The time exponent, K in the Avrami equation which governs the kinetics of recrystallisation was found to vary from 0,6 to 0,8 depending on the effective strain. It was found that the migrating boundary area S_V increases at low fractions recrystallized, goes through a maximum and decreases as the volume fraction recrystallized increases. However grain growth rates decreases with increasing

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effective time according to a relation of the form:

$$G \alpha t^{-0.72} \qquad (3)$$

In order to determine the power dependence of the recrystallised grain size on the strain applied, values of the recrystallised grain drex were taken and plotted on a logarithmic scale as a function of strains, as shown in figure 3. For the strains applied of 0.58 and 1.16, and rolling temperature of 870°C, the dependence mentioned above was found to be:

$$d_{rex} \alpha \varepsilon^{-0.6} \qquad (4)$$

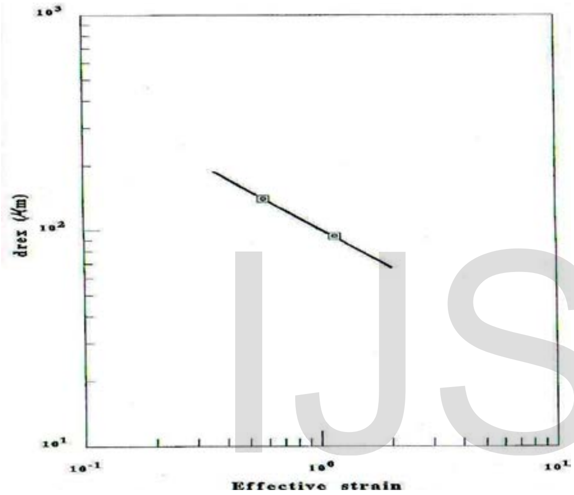

Figure 3. Effect of strain on recrystallised grain size of material of ferritic stainless steel type 409, deformed at a temperature of 870°C, d_0=160μm.

4. DISCUSSION

4.1. Structural observations

Examination of specimens quenched immediately after a single deformation pass under rolling temperatures of 870°C showed deformed grains and no recrystallisation took place following a short period of time of 4 seconds, indicating that this stainless steel also behaved as typical dynamic recovery type of metals. In this stainless steel the sample deformed at a temperature of 870°C shows that the original grain boundaries are no longer clearly visible (Figure 4), and that some larger regions contain only faint subgrains with evidence for the presence of indissolved coarse particles within deformed grains and at grain boundaries.

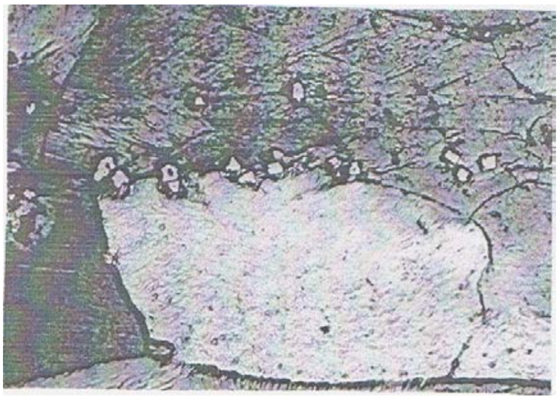

Figure 4. Optical micrograph of specimen of ferritic stainless steel type 409, deformed at an effective strain of 0,58 and at a single temperature of 870°C, showing a poorly developped substructure as well as coarse precipitates with different shapes. X300.

In stainless steel type 409 static recrystallisation is nucleated preferentially at the original grain boundary and to a lesser extent at grain edges (Figure 5). Furthermore with increasing time of holding the nucleation takes place at deformation bands.

Figure 5. Optical micrograph of specimen of ferritic stainless steel type 409, deformed at an effective strain of 1,16 and at a rolling temperature of 870°C, showing grain boundary regions as sites for nucleation. X150.

In this stainless steel which contains undissolved precipitates in the structure, the growth rate, G under the deformation condition employed tends also to decrease with time during recrystallisation and time exponent of 0.72 in equation (3) is in good agreement with the value of 0.75 reported. In practice some previous works on ferritic steels have shown that the growth rate decreased with annealing time [1,2,3,4] mainly due to stored energy consumption by recovery, according to the English-Backofen equation [2]. In one case [5] this decrease has been attributed to static recovery and in the other [2] to segregation phenomena ahead of the moving boundary. Alternative explanations for the time dependence of G has been based upon: a) Solute segregation to subboundaries in the unrecrystallised material. b) Control of the moving boundary velocity by mechanisms of desegregation. c) A time dependence in the extent of segregation [2].

4.2. Recrystallisation curves

In this stainless steel and for this fine grained material recrystallisation curves follow the Avrami-Type equation. The time exponent K was found to be equal to 0.6 and 0.8 and slightly dependent of strain, decreasing with increase in strain. The recrystallisation curves in stainless steel type 409 do not have the usual S shape. Some researchers [6] also obtained another shape of the recrystallisation curves (no incubation time) after a plane strain compression-type high-speed hot deformation simulator in the + carbide two phases regions. They attributed the appearance of this kind of curve to the increase in nucleation sites for recrystallisation due to the presence of carbides. Detailed analysis of the recrystallisation curves for the present materials suggests that stainless steel type 409 displays a slower rate of recrystallisation than stainless steel type 430 for exemple (Figure 6). It has pointed out that nitrides are all out of solution after solidification [7]. If pinning occurs it must be due to carbides. It was also pointed out that the carbides are likely to precipitate onto the preexisting nitride particles. Therefore the retardation of recrystallisation must be due to the presence of the excess elements such as Ti still in solution in the alloys.

A true activation energy for recrystallisation of $185 kJ.mol^{-1}$ for stainless steel type 409 is obtained. The value of $185 kJ.mol^{-1}$ derived in the present work is higher than the value of $120 kJ.mol^{-1}$ obtained by Craven and al [8] during recrystallisation of ferritic stainless steel which contained both Nb and Ti.

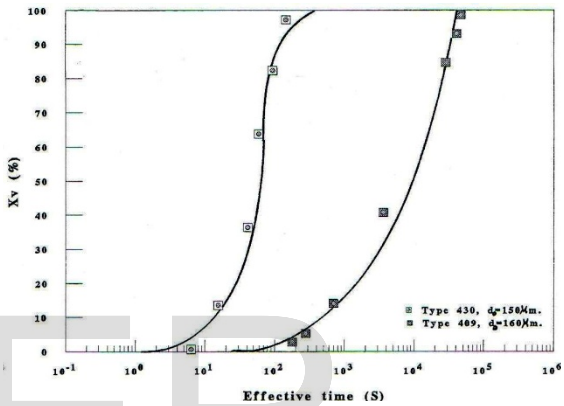

Figure 6. Isothermal recrystallisation kinetics of ferritic stainless steel type 430 and 409, deformed at an effective strain of 0,58 and at a temperature of 870°C.

4.3. Effect of strain

The effect of increasing strain leads to an increase in migrating boundary area during recrystallisation and refinement of the recrystallised grain size due to the increase in density of potential nucleation sites and the increase in stored energy of deformation. In this stainless steel the value of negative exponent of 2.8 for the dependence of $t_{50\%}$ to strains of 0.58 and 1.16, is lower than the value of 4 for steels found by many investigators [1,2,3,9,10]. Metallographically it was shown that the acceleration of recrystallisation by strain leads to more nuclei produced since the original grains continue to elongate and boundaries become serrated between subgrains.

The grain size produced after static recrystallisation is dependent on the value of the prior strain since this controls the dislocation structure and therefore the number of sites for nucleation. The higher the prior strain, the smaller is the grain size after complete recrystallisation. This dependence suggests that the recrystallised grain size is proportional to a strain exponent of - 0.6. A strain exponent of -1 has often been reported for ferritic and austenitic steels [3,11,12].

4.4. Effect of particles

In our case, the type 409 alloy (0.016% carbon and 0.32% titanium) in the temperature of 870°C contains titanium giving rise to yellow coarse, angular non-metallic particles, observed by optical microscopy and probably correspond to $Ti(C_XNy)$. These particles appear to be responsible for the retardation of recrystallisation. Coarse particles may create local lattice distortion in the matrix around the particles, if a critical strain rate and temperature condition is exceeded [13,14]. These distorted regions cause high local dislocation densities, which may result in particle stimulated nucleation of subsequent static recrystallisation, with important effects on both the kinetics of recrystallisation and the texture developed [15]. These yellow coarse particles ($Ti(C_XNy)$) seem to affect recrystallisation indirectly through their influence on the preceding recovery processes and on moving grain boundaries since nucleation sites were observed only at grain boundaries. Whatever the cause, it is clear that the retardation of static recrystallisation in this stainless steel is attributable, in a large measure, to the retardation of recovery caused by the titanium addition. However, titanium is known to retard recrystallisation kinetics, whether in solid solution or precipitate form [16].
In general, recrystallisation is controlled by precipitation of carbonitrides and clearly variables such as reheating temperature, which determines the solution rates of carbonitrides, amount of deformation/pass, which affects the amount and mode of precipitation of carbonitrides, and finish rolling temperature, which alters the amount of precipitation on further cooling, must be carefully controlled.

5. CONCLUSION

The dynamic restoration process which operates during hot working at a constant strain rate of $3,3s^{-1}$ and temperature of 870°C is dynamic recovery.
In this stainless steel type 409, TEM showed that lower temperature of deformation (870°C) produce ragged subboundaries with subgrain interior more densely populated by dislocations and at lower temperature of deformation (870°C), the original grain boundaries are no longer visible and some larger regions contain only faint subgrains. There is evidence of the presence of coarse titanium carbonitrides Ti(CN) within deformed grains and at grain boundaries.
In this stainless steel type 409, nucleation of recrystallising grains appears to be associated with triple points and grain boundaries and no evidence of nucleation at particles of titanium carbonitrides was observed. The growth rate of recrystallising grains decreases with time of annealing according to a power relationship with an exponent of (-0,75) and it is found to be in good agreement with the ones previously reported in the literature. The recrystallisation kinetics follow Avrami behaviour. The true activation energy for recrystallisation is $185kJ.mol^{-1}$.
Stainless steel type 409 displays a slower rate of recrystallisation than stainless steel type 430 and this is attributed to the presence of titanium. The recrystallised grain size seems to be independent of reheating temperature.

REFERENCES

[1]. G.Krauss and S.W.Thomps: ISIJ International. Vol.35 (1995). No.8, PP. 937-945.

[2]. D. J. Jensen: Scripta METALLURGICA et MATERIALIA. Vol.27, PP.533-538, 1992.

[3]. A.M.J.Júnior and O.Balancin: Rev. LatinAm. Met.Mat. V.22. n.1. Caracas ene. 2002.

[4]. S.B.Singh and H.K.D.H.Bhadeshia: 'Topology of grain deformation', Materials Science and Technology, 1998, 15, 832–834.

[5]. H.J.McQueen, Thermomechanical Processing of Steel (Jonas Symposium), 2000, S.Yue, E.Essadiqi, eds., Met.Soc. CIM, Montreal, PP. 323-333.

[6]. G.Lefebvre: Ph.D Thesis, University of british columbia (Vancouver), 2014.

[7]. R.G.Nooning, Jr: Masters of Science Thesis, University of Pittsburgh, 2002.

[8]. A.J.Craven, K.He, L.A.J.Garvie and T.N.Baker: Acta Mat. Vol48 (2000). PP.3857-3868.

[9]. J.D.Fritz and I.A.Franson, "Sensitization and stabilization of type 409 ferritic stainless steel," Materials Performance, Vol.36, No.8, PP.57–61, 1997.

[10]. T.Gladman, The Physical Metallurgy of Microalloyed Steels, Institute of Materials, London, England, 1997.

[11]. T.Gladman, The Physical Metallurgy of Microalloyed Steels, Institute of Materials, London, England, 1997, P.48.

[12]. R.A.N.M.Barbosa and C.M.Sellars: Materials Science Forum, 1993, 113-115, P.461-466.

[13]. R.Moses, A.Zarei.Hanzaki, S.Yue and J.J.Jones, 35th MWSP Conf.Proc., ISS-AIME, Vol.XXXI, 1994, PP.435-442.

[14]. R.G.Nooning, and T.Denardo, 'Precipitation and stabilization of T409 Ferritic Stainless Steels, Senior Thesis, Department of Materials Science and Engineering, University of Pittsburgh, 1999.

[15]. D.Raabe and K.Lucke, 'Textures of Ferritic Stainless Steels', Materials Science and Engineering, Vol.9 (1993), PP.302-312.

[16]. B.Feng, D.Dunne and T.Chandra: The Minerals, Metals and Materials Society, 1990, P.319-324.