Hot forming of duplex stainless steels

M. Barteri, E. Evangelista, H. J. McQueen, B. Verlinden

The duplex stainless steels (DSS) have yield strength and corrosion resistance superior to austenitic (γ-ss) and ferritic (α-ss) stainless steels. In hot working, the DSS high ratio mixture of γ particles in α matrix has greater strength than either phase because of mutual slip constraints hardening both phases and phase morphology retarding the normal restoration mechanisms.
The temperature dependence is greater because there is a tendency for the soft α phase to increase in addition to each phase softening. The α phase undergoes dynamic recovery (DRV) but to a diminished degree, developing a small frequency of high misorientation walls and undergoing static recrystallization on slow cooling. At high temperature the γ phase dynamically recrystallizes but this is retarded to high strains by the lack of grain boundaries in the small phase regions.
Ductility is lower than either independent phase since the interphase cracking cannot be hindered by triple junction accommodation by DRV as in the α phase or by crack isolation by boundary migration in DRX as in the γ phase.

INTRODUCTION

Duplex Stainless Steels, containing austenite and ferrite, are a class of structural materials widely used since they combine the properties of the fully ferritic or fully austenitic steels exhibiting performances not attainable with either single phase. In fact toughness and weldability of the austenite are synergetically combined with the strength and corrosion resistance of the ferrite. As a result these steels combine high strength (yield strength ≥ 450 MPa in the annealed condition and in superduplex 550-630 MPa) and good toughness behaviour, (KCV at least 75 J/cm^2 at -30°C), combined with outstanding resistance to various types of corrosion (stress corrosion cracking, pitting and crevice).
Along the years, these properties received substantial improvements from continuous progress in the steel making processes (vacuum or argon degassing). Moreover drastic reduction of oxygen, sulphur and carbon levels, tight control of chemical composition, the addition of nitrogen as structure stabiliser and of copper as microstructure refiner were achieved. The decrease of residuals has improved the hot working behaviour allowing the production of wide gauge plates and coils. Such features, together with high cost effectiveness from the low Ni content, explain their increasing demand in the chemical, oil and gas industries, off-shore structural systems, chemical tankers, containers for food production, heat exchangers and many other applications where the corrosion resistance to a variety of aggressive environments and chemical products and strength greater than that of austenitic or ferritic stainless steels are the main concerns [1].
Many grades of duplex stainless steels in the form either of wrought products or castings are available. At present for the wrought products, there are three principal grades with chemical compositions between the limits: Cr 22-25%, Ni 3-7%, Mo 3-4%, N 0.10-0.17%, these confer on the steels the required corrosion and mechanical properties. Usually the alloys have about equal volume fractions of ferrite, α, and austenite, γ, obtained by hot working, solution annealing and quenching. An important parameter for corrosion and hot working behaviour, is the "partition coefficient" between α and γ phases taking into account that the ferrite is enriched in a stabilising elements P > W > Mo > Cr ≅ Si and austenite in γ stabilising N >> Ni > Cu > Mn; the elements are in sequence of decreasing stabilising effectiveness [2].
Whereas the hot forming behaviour of austenitic and ferritic stainless steels in hot torsion, tension and compression tests has been widely studied and results analysed thoroughly in several review papers [3,4], few data are available on DSS. In comparison to austenitic and ferritic stainless steels the duplex ones are more sensitive to process parameters (T, $\dot{\varepsilon}$ soaking time) since ferrite and austenite have different crystal structure and as a consequence different response to restoration occurring during and after deformation.
A strong dependence of hot working characteristics on volume fraction of α phase was pointed out by Jervinen [5] studying by tensile, compression and rolling several DSS at various T and strain rate. The increase of α produced localised deformation in γ while raising ductility and depressing the strength. As will be discussed fully later, Chandra and colleagues [6] and Al-Jouni and Sellars [7] studied the intertwined problems of partitioning of strain; the mutual strengthening effects and the restoration mechanisms in each phase which are inherently very different as explained in the next section. Verlinden and co-workers [8] showed that N additions raise the flow strength by strengthening the γ phase and stabilising it in higher volume fraction.
Hot compression of as cast 2205 DSS and AISI 304 have been investigated in the ranges 1000-1250°C and $\dot{\varepsilon}$ = 0.1-10 s^{-1} by Paul and co-workers [9]. The flow stress had the same trend for both but with lower values in the DSS due to the presence of 65% soft ferrite. The effect of temperature and strain rate on strength and ductility of 2304 DSS was investigated by means of hot torsion tests by Barteri et al. [10]. The peak stress and the ductility were influenced by α / γ ratio and by strength of the phases modified by addition of Mo and N. DRV of ferrite at all temperatures and DRX of austenite above 1100°C were found to be the active softening mechanisms. The softening of ferrite in 21.10 DSS deformed in torsion at $\dot{\varepsilon}$ of 0.7 s^{-1} at 1200°C was investigated by Cizek and Wynne [11]. Quantitative optical microscopy and TEM determination of the misorientation across α/α boundaries affirmed that ferrite softening was based on extended DRV without occurrence of discontinuous DRX.
This paper aims to present and discuss the results on hot-forming behaviour of some relatively novel DSS used from the beginning of the present decade.

M. Barteri, *Centro Sviluppo Materiali, Roma, Italy*
E. Evangelista, *INFM, Dipartimento di Meccanica, Università di Ancona, Ancona, Italy*
H. J. McQueen, *Mechanical Eng., Concordia University, Montreal, H3G 1M8 Canada*
B. Verlinden, *Katholieke Universiteit Leuven, Heverlee, Belgium*

Paper presented at the International Conference Stainless Steel'99, Chia Laguna, 6-9 June 1999, organised by AIM

HOT DEFORMATION OF FERRITE AND AUSTENITE.

A fundamental stage in understanding the high temperature deformation of mixed α and γ phases is knowledge of the deformation of ferrite and austenite separately. This information is copiously available since the hot workability of ferrite has been studied in pure Fe, Fe-Si and Fe-Cr stainless steels [12-14]. High temperature deformation of austenite is widely used in the normal processing of C, HSLA and tool steel and also of the austenitic stainless steels. Both flow stress dependence on T and ε and microstructural evolution are available for both phases. However the phase transformation on cooling of C, HSLA and tool steels makes it impossible to examine the dislocation substructures which are observable in γ stainless steels. Before presenting details, one needs comment on the results showing that the mechanisms of restoration in the two phases are at the extremes of the behavioural spectrum dynamic recovery, DRV, in ferrite and DRX in austenite.

Ferritic steels provide a good example of a high degree of DRV in which the dislocations are able to cross-slip and climb in order to annihilate and to rearrange into a polygonized subgrain structure [12,14-19]. The subgrains remain equiaxed, constant in size and misorientation to strains exceeding 4 which are higher than those in most forming processes. As a result of developing constant dislocation density, the flow curve rises monotonically to a steady state plateau (σ_s, $\varepsilon > \varepsilon_s$). As temperature rises and strain rate falls, the subgrain size increases, dislocation density decreases and the steady state flow stress decreases. In many cases, the steady state regime exhibits a gradual decline due either to deformation heating which is greater for higher stress and work or to microstructural evolution such as the particle size and distribution. The high level of DRV leads to a high ductility since the low flow stress leads to low stress concentrations at grain boundaries reducing crack initiation [12,16]. The change in properties and mechanisms from cold working to temperatures above 500°C is gradual and permits so called warm forging or rolling with forming forces reduced and ductility raised [12,16,20].

In marked contrast to the above, austenitic steels, which have low stacking fault energy (SFE) limiting dislocation climb and cross slip, have a much lower level of DRV [4,12,19,21-30]. The rate of strain hardening is high in association with a rising dislocation density until a critical condition (σ_c, ε_c) is reached at which new grains are nucleated and spontaneously grow during straining in the mechanism called DRX [24,30]. The rapid elimination of dislocations by the growth of new grains in necklaces along the grain boundaries (for suitable combinations of high strain rate and large initial grain size) leads to a flow curve peak (σ_p, ε_p) and rapid work softening (≈20%). This is followed by a steady state regime (σ_s, ε_s) in which a constant substructure is maintained by a combination of DRV and DRX yet at a high flow stress compared to ferrite under the same conditions. Good ductility arises from DRX since the migrating grain boundaries impede the nucleation and growth of cracks to give a ductility usually less than that of ferrite [4,12,14,31].

Both mechanisms are affected by alloy additions but usually the austenite to a much higher degree. Solutes tend to decrease dislocation mobility and reduce DRV in both materials. Small particles serve as barriers to dislocation motion and also decrease DRV. These effects raise the flow stress, the stress concentration and thus augment GB cracking. The presence in austenite of segregated solute and of small to medium particles in high density can impede boundary migration and hence slow down DRX and drastically reduce ductility [12,13,31-33]. The 300 series stainless steels with solute levels in the range 25-40% clearly exhibit marked solute strengthening [24,26]. The presence of non deforming large particles or inclusions can develop voids especially in less ductile or higher strength alloys, leading to rapidly propagating cracks; austenite is thus more susceptible to reduction in hot workability than ferrite. In higher carbon steels the C dissolved in the austenite expands the lattice and enhances DRV whereas in the ferrite it leads to formation of carbides which raise strength and reduce ductility dependent on the morphology [4,14,33,34]. Of course alloy carbides in tool steels at 900-1200°C raise strength and lower ductility [34].

The static softening after any stage of deformation is also much affected by the level of dislocation density left by the restoration mechanism [4,12,14,33]. The high level of DRV in ferrite usually leads to gradual softening as a result of static recovery SRV so there is very little softening and grain refining between passes. This makes it easy to retain the deformation substructure to strengthen the product but it does not provide a soft fine grained product for cold forming [12,14,16]. On the other hand the high dislocation densities in austenite lead to rapid static recrystallization with grain refinement and loss of all deformation strengthening [12,27,28]. In multistage processing of austenite at high temperatures this is exploited to eliminate the as cast structure and improve the ductility [13,32,35,36]. In combination with reduced rates of recrystallization at low T (≈ 850°C) in C and HSLA steels, it is expected in controlled rolling to produce a high grain boundary area in the austenite, through refinement or pancaking in order to control the size of the ferrite grains [4,27,33].

HOT STRENGTH OF DUPLEX STAINLESS STEELS

The hot strength of metals is an important technological parameter since it controls the forming forces and work; it is controlled by the balance between work hardening and softening from restoration processes. The dependence of hot strength on temperature and strain rate and on internal factors such as stacking fault energy, solute content as well as volume fraction, composition, size and shape of second pha-

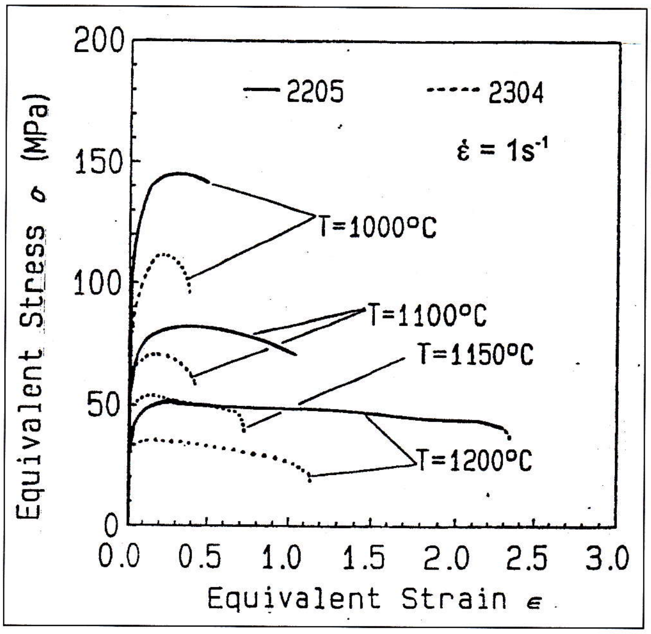


Fig. 1. Equivalent stress-equivalent strain curves of two DSS designated as 22.05 (22 Cr, 5.3 Ni, 3 Mo, 0.16 N, wt%) and 23.04 (22 Cr, 4 Ni, 0.2 Mo, 0.1 N, wt%).

Fig. 1. Curve di tensione equivalente-deformazione equivalente degli acciai duplex: 22.05 (22 Cr, 5,3 Ni, 3 Mo, 0,16 N, peso%) e 23.04 (22 Cr, 4 Ni, 0,2 Mo, 0,1 N, peso%).

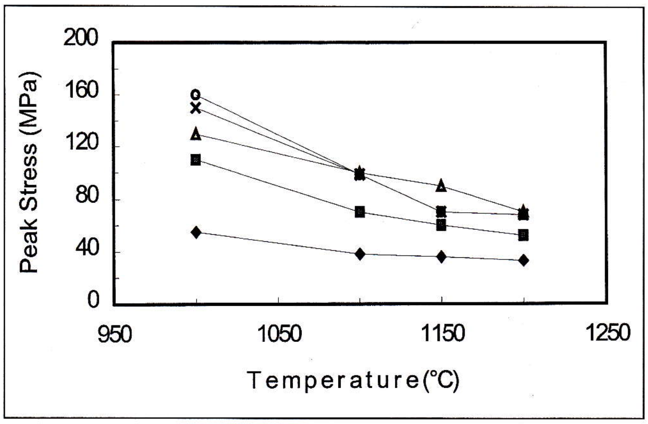


Fig. 2. Peak stress as a function of deformation temperatures: μ 27.04 [8], 6 22.05 [10], σ AISI 304 [18], v 23.04 [10], and υ AISI 430 [19].

Fig. 2. Tensione di picco in funzione delle temperature di deformazione: μ 27.04 [8], 6 22.05 [10], σ AISI 304 [18], v 23.04 [10], and υ AISI 430 [19].

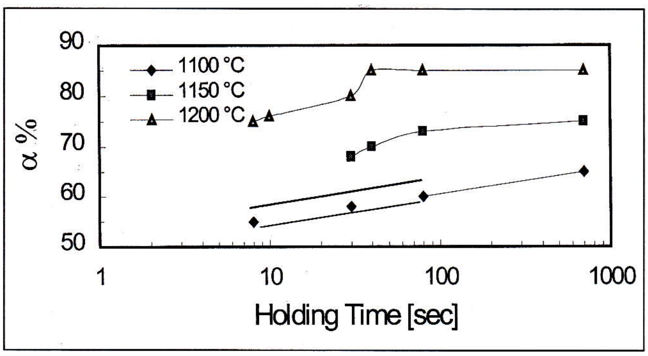


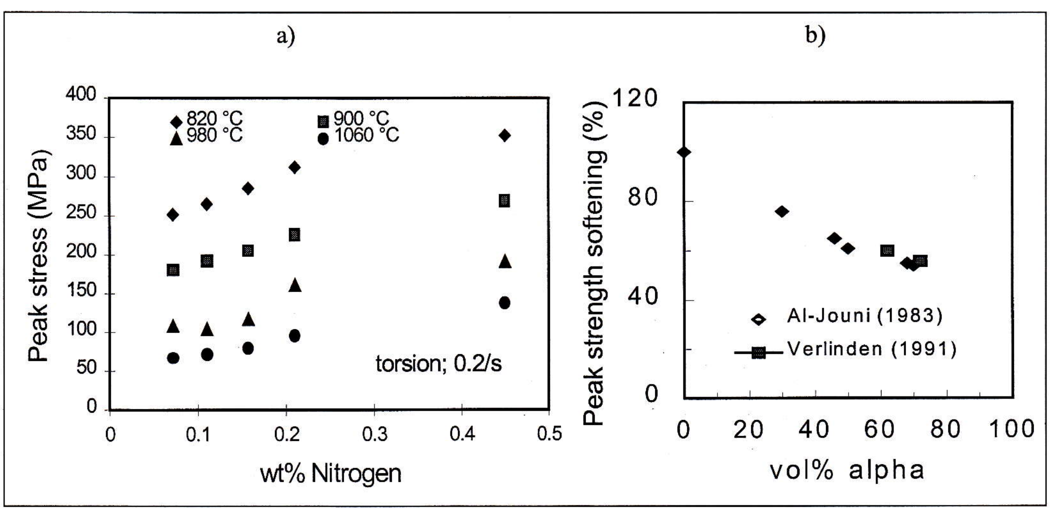
Fig. 3. Effect of holding time on content of α phase at various temperatures in 23.04.

Fig. 3. Effetto del tempo di permanenza a varie temperature sul contenuto della fase α in 23.04.

ses is well documented [3,4,12,14,16,33]. For a duplex structure of different strengths, the volume fraction of each phase at the working temperatures is very important. The δ ferrite, produced by solidification of DSS, transforms, on cooling, to austenite; since the transformation is reversible, the temperature variations affect the volume fraction and the chemical composition of the two phases.
The high temperature mechanical behaviour of 22.05 and 23.04 were determined by torsion testing. Some representative flow curves of the two DSS in Fig.1 show hardening to a peak and a progressive softening to a steady state. The trend of stress peaks versus temperature at constant strain rate are shown in Fig. 2 [8,10,15,24]. As compared to α or γ steels, the decrease in peak stress in DSS with increasing temperature is enhanced by the increase in the volume percent of ferrite. However, if the pre-treatment before torsion is carried out in such a way that the volume percent of ferrite remains constant at all test temperatures, a normal temperature dependence of DSS is observed [8]. Because α is the matrix phase, rising α means less strengthening γ particles. When γ is the matrix, 10% δ-phase in as cast 304 raises the strength at 900°C, 5 s^{-1}, to 250 from 200 MPa in the homogenized. This occurs because δ causes slip constraint while suffering the same. Such δ particles produce a denser dislocation substructure in the surrounding γ phase leading to particle stimulation of DRX or SRX nucleation [22,23,32].
Al-Jouni and Sellars [7] studied in detail the effect of increasing content of δ ferrite on flow peak, activation energy and partitioning strain between the phases. In deforming by torsion and rolling in the interval 900-1200°C some DSS with a content of δ ferrite 0, 30, 50, 70, 100% and constant solute level, they found that flow stresses and activation energies for deformation varied with volume fraction of the phases but not linearly following the law of mixtures based on equal strain in the two phases. The activation energy varied from 400 to 340 kJ/mol passing from 100% of austenite to 100% of ferrite. Considering the strain partition effect, both phases became equally deformed under medium strain when δ is > 30%. In these conditions the austenite underwent DRX following kinetics as in single phase austenitic steels.
The effect of N on hot strength was analysed by Verlinden [8] who tested in torsion some DSS with approximately constant solute content (about 29%) but with N varying from 0.07 to 0.4 wt%. For temperatures from 1000 to 1300°C, ferrite volume fraction is stable for N = 0.25-0.4%, but for lower N content (< 0.18%) the phase increases dramatically up to 85% at 1300°C [1]. The influence of holding time on γ→α transformation is shown in Fig. 3 [1]. In the interval 800-1060°C, an apparent strengthening effect of 270 MPa / wt% N was observed, Fig. 4a. This effect is partially due to an inherent strengthening and partially to a decrease of ferrite with increasing N that is a γ-stabiliser. Based on the results of Al-Jouni, Verlinden used a "calibration curve", Fig. 4b [8] which permits evaluation of the influence of a change in ferrite content and to recalculate the experimental peak stresses to hypothetical stresses at constant ferrite content. In Fig. 5, the experimental data obtained at 900°C in torsion with amount of ferrite ranging from 57 vol.% at 0.07 wt% N to 39 vol.% at 0.40 wt% N, have been recalculated to constant levels of either 57 or 39 vol.% a.
In this way the inherent strengthening effect of N could be

Fig. 4. Influence on peak stress of: a) the N amount, b) the ferrite content of different steels.

Fig. 4. Influenza sulla tensione di picco: a) del peso% di N e b) del contenuto della ferrite .



Memorie

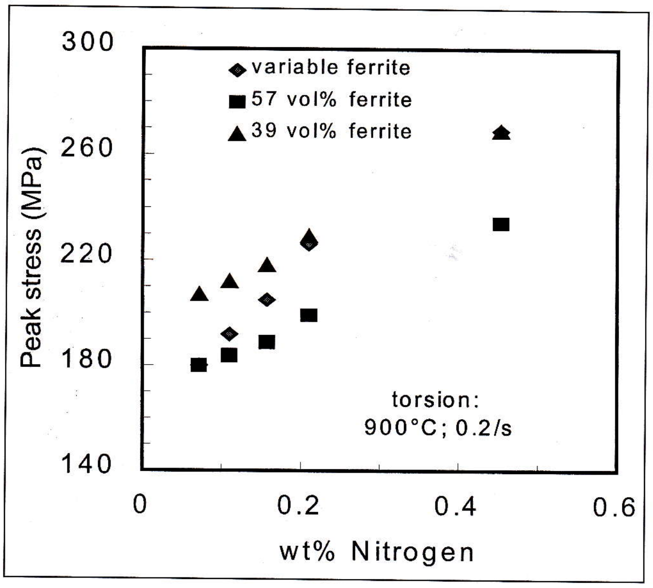


Fig. 5. Calculated peak stresses for constant αvol% fractions of 39 and 57.

Fig. 5. Tensioni di picco calcolate a frazioni in volume di fase α rispettivamente del 39 e 57 %.

evaluated and is about 130 MPa/wt% N. The beneficial effects of nitrogen-alloying on austenitic stainless steels has been discussed in many conferences, particularly in the HNS (High Nitrogen Steels) ones and in a recent review paper of Simmons [37]. In DSS the nitrogen addition improves resistance to localised corrosion, strength and stability of the two phase structure [1].
The influence of grain size on the peak stress was considered by Verlinden and co-workers [8] in the AISI 316N type steel in which the increase of grain size reduced both the ductility and the peak stress. In the DSS investigated only a 5% decrease of the peak stress with increasing grain size was observed. As far as the distribution of strain between the two phases is concerned, in hot deformation of the DSS Sandvik 3RE60 at $\dot{\varepsilon}$ = 0.0038 s^{-1} and strain of 0.023, a polygonized structure appeared only in the α phase. Since dynamic recovery appeared in γ phase only at strain >0.38, Chandra et al. [6,38] concluded that γ was more resistant to straining and that the total strain was not distributed homogeneously into the two phases but was directed, in the main part, to deform the α matrix. The conclusion was also confirmed by optical microscopy observation that the γ particles began changing shape at strain 0.49. Such distribution of the strain in the two phases agrees with observations of Al-Jouni and Sellars [7].

HOT DUCTILITY OF TWO PHASE ALLOYS

The torsional ductility, ε_f, of 22.05 as T rose from 1000 to 1200 °C with almost γ constant of 70%, increased from 0.7 to 2.9 at $\dot{\varepsilon}$ = 0.1 s^{-1} and 0.6 to 1.2 at $\dot{\varepsilon}$ = 5 s^{-1}, table 1.
Over the same T range with γ declining from about 40 to 22%, ε_f of 23.04 varied from 0.4 to 1.2 at 0.1 s^{-1} and 0.4 to 0.7 at 5 s^{-1}. Similarly as cast 304, with about 10% segregated δ, exhibited ε_f from 1.0 to 1.2 (0.1 s^{-1}) and 0.9 to 1.0 (5 s^{-1}) with cracks always originating at γ–δ interfaces [13,19,22,23,31]. In contrast, homogenized 304 showed ef of 5.7 to 11.8 (0.1 s^{-1}) and 6.5 to 13.4 (5 s^{-1}) (at higher ε, ε_f declined) with enhanced DRX slowing fissure growth [4,12,24,30].
The interaction of the two phases has been studied in the intercritical α–γ range of C and HSLA steels since it marks the lower limit of commercial hot working processes. The appearance of ferrite grains along the austenite grain boundaries can lead to a marked decrease in ductility especially if they form on large equiaxed austenite grains during the preheating. On the other hand in controlled rolling where the α gradually forms between successive passes on the boundaries of the pancaked austenite grains, the ductility may not be much reduced [12,27]. The ductility may rise and the flow stress decline since the bands of ferrite have good dynamic softening and do not inhibit softening in the austenite to a harmful degree. They strengthen the product ferrite in providing strain hardened layers.
In alloy steels notably tool and stainless steels, the two phase structures can also be studied in the γ–δ intercritical region. If δ appears due to over heating it may have deleterious effect on the austenite by impeding DRX. If γ forms in the δ phase on cooling the effect is not severe since the hardening is in proportion to the volume fraction and the DRV mechanism is not impeded nearly as much as similar particles in austenite would prevent boundary migration [4].

MICROSTRUCTURES AND MECHANISMS

As seen in optical micrographs, Fig. 6 a,b,c, the austenite occurs as smoothly irregular particles of about 20-50 μm ranging from equiaxed to much elongated (≈300μm) in a ferritic matrix with interparticle spacing of about 20-150 μm. After extended heating and temperature cycling, the γ particles become coalesced, i.e. more uniform and spheroidal and smaller as T is raised above 900°C. In deformed and quenched specimens, the a phase contains subgrains more clearly defined as e rises above 0.5 and larger as T increases and $\dot{\varepsilon}$ decreases. The γ phase etches only to exhibit grain boundaries and straight annealing twins crossing some elongated particles. With holding of 20-80 seconds after deformation, the α phase exhibits enlarged subgrains and in some regions large SRX grains with the GB extending across the phase layer; the γ exhibits an increased frequency of annealing twins as if SRX was more general, each particle being one grain. This is in agreement with the determination by Urcola and colleagues [39] of SRX in the γ phase with only SRV in the α phase of two DSS 23Cr-4.8Ni-0.22Mo-0.095N (50% γ) and 22Cr-5.2Ni-0.17Mo-0.18N (75% γ) after plane strain compression in the ranges 930-1100°C and 0.1-3 s^{-1}. The γ phase undergoes DRX but likely at much higher critical strain than in the massive phase (for similar T, $\dot{\varepsilon}$) because

	1000°C				1100°C				1150°C		1200°C			
	22.05		23.04		22.05		23.04		23.04		22.05		23.04	
$\dot{\varepsilon}$ (s^{-1})	71.5% γ		40% γ		72.5% γ		35% γ		31% γ				22% γ	
	σ_p (MPa)	ε_f	σ_p (MPa)	ε_f	σ_p (MPa)	ε_f	σ_p (MPa)	ε_f	σ_p (MPa)	ε_f	σ_p (MPa)	ε_f	σ_p (MPa)	ε_f
0.024	62	1.2			42	2.1					18	1.0		
0.1	92	0.7	82	0.4	50	1.8	48	0.6	36	0.9	32	2.9		1.2
1.0	146	0.9	110	0.4	82	1.0	65	0.5	55	0.7	54	2.2	38	1.13
5.0	176	0.6	123	0.4	104	0.6	90	0.6	65	0.7	72	1.2	56	0.7

Table 1. Peak strength and ductility dependence on T, $\dot{\varepsilon}$ and vol% γ phase.

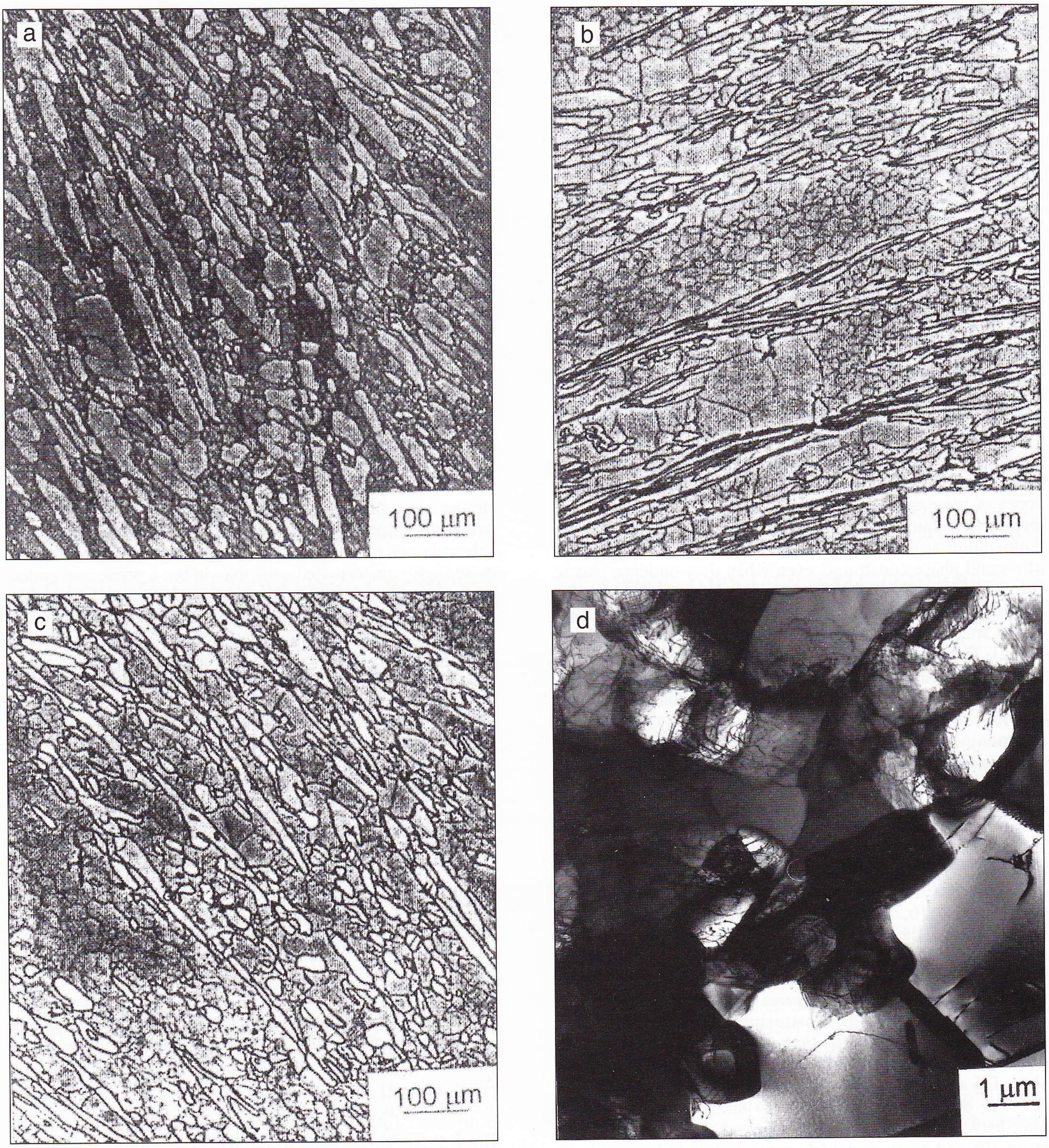


Fig. 6. Optical (a,b,c) and TEM (d) micrographs of 22.05 after hot torsion tests at: 1000°C, ε = 0.3, 0.1 s^{-1} a); 1200°C, ε = 0.9, $\dot{\varepsilon}$ =0.1 s^{-1} b); 1200°C, ε = 0.3, $\dot{\varepsilon}$ = 5 s-1 c and d).

Fig. 6. Micrografie ottiche (a,b,c) ed elettroniche in trasmissione dell'acciaio 22.05 dopo prove di torsione a 1000°C, ε = 0.3, 0.1 s^{-1} a); 1200°C, ε = 0.9, $\dot{\varepsilon}$ =0.1 s^{-1} b); 1200°C, ε = 0.3, $\dot{\varepsilon}$ = 5 s-1 c ed).

GB serve as the nucleation sites for the necklace structures commonly responsible for grain refinement [24,30], (equilibrium DRX grain size is often larger than the particle size in DSS). The DRX necklaces normally halt GB cracking in γ-SS or in γ-Fe but are ineffective against interphase cracking [4,12,31]. At fracture in the ε range 0.7-1.5, the phases appear only slightly elongated.

As observed by TEM, the two phases are easily distinguished by the larger more polygonized subgrains in the α phase as well as by diffraction. Because of the irregular phase shapes and small dimension in one direction, it is difficult to observe gradients in cell dimensions. However in each phase there are considerable variations in cellular dimensions and dislocation density from region to region depending on phase morphology, Fig. 6d.

A recent study by Cizek and Wynne [11] on 21Cr-10Ni-3Mo confirms these features and indicates DRX of the γ phase near the peak of the flow curve ($\varepsilon_p \approx 0.5$, $\sigma_p \approx 50$ MPa, 1200°C, 0.7 s^{-1}). The recovered substructure in the α phase was studied intensively by selected area diffraction and maps of boundary misorientation determined.

In addition to a small number of original GB, the average

misorientation, $\psi \approx 5°$ with about 10% spread across the interval 11-19°. The origin of some of these high ψ walls is undoubtedly disorientation boundaries DB between deformation bands that arise from the grains splitting into regions slipping on different systems according to Taylor theory. Moreover DB are permanent, not continually rearranging like sub boundaries, so that they increase in misorientation with strain [40-43].
DRX seems to be ruled out but the high ψ indicate that the DRV is not ideal as in creep but is characteristic of warm (wDRV) as occurs in hot working where T decreases and $\dot{\varepsilon}$ rises in the range approaching warm working as discussed extensively in Al-alloys [41-43]. These results confirm the observations of recovery in ferritic alloys by McQueen, Evangelista and Ryan [17-19], by Lombry et al. [15] (maximum $\psi \approx 13°$ at $\varepsilon = 2.7$), by Schimdt et al. [44] (mean ψ rising to 15° at $\varepsilon = 15$) and by others [22-23]. This is a different interpretation from DRX proposed by Kaibyshev and others [44-46] and disputes the occurrence of DRX suggested by a few [47-51].
The transition from ideal hot working or creep DRV to wDRV is induced not only by falling T and rising $\dot{\varepsilon}$ but also by the presence of fine particles which develop surrounding zones of increased dislocation density. Solutes which lower the stacking fault energy or cause solute drag can raise the temperature at which the transition takes place. The effects of second phase constraints even when they undergo plastic flow notably on totally different slip system result in increased strain hardening and higher flow stress (thus wDRV); this is true even of δ phase particles in austenite [8,12,22-24,32]. The mutual strengthening of both phases can produce strengths higher than the rule of mixtures. In addition from such effects, reduced cell size in the δ phase relative to that in single phase ferrite does not confirm that strain is partitioning to it excessively. The interphase cracking which is the source of failure is indicative of the slip constraints (even δ in γ) and the interface stress concentrations. The limited interphase spacing and volume fraction gives no opportunity for lattice accommodation at triple junctions in the a which normally stops GB cracking.
In a 18.05 type DSS, thermally treated and quenched in order to produce 50% α in the form of Widmanstatten plates, Chandra et al. [6,38] found that at 900°C and at strain rates up to 0.038 s^{-1}, the flow stress increases to strain of 0.5 with α phase dynamically recovered in well defined subgrains at e > 0.1, whereas g starts to recover at $\varepsilon > 0.4$ in cells with tangled boundaries. Strain appears non uniformly distributed since α exhibits more substructure than γ and finer than normal. Only at 0.38 s^{-1}, the flow curves show a peak typical of DRX; but new grains appeared only in γ close to α phase at $\varepsilon = 0.6$ and imperfect subgrains are formed in the remainder. The anomaly was attributed to unequal strain partitioning between the two phases. At lower strain rate, the a is more affected by strain which transfers to g at higher strain rates producing a critical strain to induce DRX [12].
The effects of two phase structures have also been examined in as-cast austenitic stainless steels containing segregated δ ferrite to which ferrite stabilising elements are positively segregated and austenite stabilizers negatively [12,21-23,32]. Although the ferrite is inherently softer, its presence in an elongated network blocks slip transfer between large austenite grains greatly increasing strain hardening and thus raising the peak stress in the flow curve compared to the homogeneous alloy. The increased dislocation density and reduced cell size around the particles stimulates nucleation (PSN) of DRX (or SRX after straining). Although DRX is speeded up, fissures are nucleated at the γ – δ interfaces which leads to very low ductilities relative to the homogenized alloys [13,21,23,31]. The δ ferrite particles exhibit recovered substructures. In analogy to this, the presence of segregated γ phase in a Widmanstatten like structure in as cast ferritic stainless steels leads to an increase in strength and a noticeable peak and work softening towards the levels of homogenized alloys as the γ phase becomes less able to block DRV. The ferrite phase develops a recovered substructure and does not undergo DRX even in the vicinity of the austenite particles [17-19].

CONCLUSIONS

The DSS have higher strength than expected from the rule of mixtures of the α and γ phases as a result of mutual strengthening due to slip constraint increasing dislocation density in both phases. The decrease in strength with rising temperature is generally greater than in austenitic or ferritic steels because of the increase in ferrite volume fraction but is similar when phase fractions are constant. There is some evidence of strain partitioning preferentially to the softer α phase at low strains and volume fractions; this conclusion may be questioned because of the greater capability in observing substructural changes in a than in γ phases. The α phase develops well defined subgrains after relatively low strains but at high $\dot{\varepsilon}$ some large misorientations probably result from deformation band rotation. The γ phase develops substructures of much higher dislocation density in smaller more highly misoriented cells than in the a phase and undergoes discontinuous DRX at critical strains much higher than in simple γ-SS due to the absence of nucleation sites at internal grain boundaries within the γ particles. The DRX in the γ phase is commonly the cause of the peak in the flow curve but does not induce DRX in the α phase. Upon annealing, the α phase undergoes considerable static recovery before recrystallizing whereas the γ phase particles recrystallize readily to single crystals traversed by annealing twins.

ACKNOWLEDGEMENTS

The authors (E.E and M.B) wish to thank Centro Sviluppo Materiali, Roma, funding that has made the collaboration possible.

REFERENCES

1) J. CHARLES, Duplex Stainless Steels, J.Charles and S.Bernhardsson eds. Les Editions de Physique, Paris (1991). p 1.
2) J. CHARLES, Innovation Stainless Steels, AIM, Milano (1993). vol. 3, p. 29.
3) B.AHLBLOM and R.SANDSTROM, Int. Metall.Review, 27, (1982). p.1.
4) H.J.McQUEEN and J.J.JONAS: J.Appl.Metal Working, 3 (1984-85). p.233 and 410.
5) P.A.JERVINEN, Proceeding 4th Nordic High Temperature Symposium, Helsinki (1975). vol.2, p.137.
6) T.CHANDRA, D.P.DUNNE and P.CAMPBELL: Strength of Metals and alloys (ICSMA6). R.C.Gifkins, ed. Pergamon Press, Oxford (1983). vol.1, p. 505.
7) F.E.AL-JOUNI and C.M.SELLARS, Deformation of multi-phase and particle-containing materials. Bilde-Sorensen et al eds. Roskilde (1983). p. 131.
8) B.VERLINDEN, K.CUYPERS and E.AERNOUDT, Duplex Stainless Steels. J.Charles and S.Bernhardsson eds. Les Editions de Physique, Paris (1991). vol.2, p. 711.

9) A.PAUL, J.L.MARTOS, R.SANCHEZ, Innovation Stainless Steel. AIM, Milano (1993). vol. 3, p. 297.
10) M.BARTERI, E.EVANGELISTA, M.NIEWCZAS and L.SCOPPIO, Innovation Stainless Steel. AIM, Milano (1993). vol.3. p. 285.
11) P. CIZEK and B.P.WYNNE, Mat. Sci. Eng. A230 (1997). p. 88.
12) H.J.McQUEEN, N.D.RYAN, R.ZARIPOVA and K.FARKHUTDINOV, Proc.37th Mechanical Working and Steel Processing. Iron and Steel Inst. AIME, Warrendale (1996). p. 883.
13) E.V.KONOPLEVA, H.J.McQUEEN and N:D.RYAN, Hot Workability of Steels and Light Alloys Composites. H.J.McQueen et al. eds. Met.Soc. CIM, Montreal (1996). p. 431.
14) H.J.McQUEEN and D.L.BOURELL, Formability and Metallurgical Structure, A.K.Sachdev and J.D.Embury eds., TMS, Warrendale (1986). p.341.
15) R.LOMBRY, C.ROSSARD and B.J.THOMAS, Rev. Met. 78, (1981). p.975.
16) D.L.BOURELL and H.J.McQUEEN, J.Mat Shaping Tech. 5, (1987). p. 53.
17) E.EVANGELISTA, J.BOWLES and H.J.McQUEEN, High Temp.Mat.Proc. 12, (1993). p. 57.
18) H.J.McQUEEN, E.EVANGELISTA, P.MENGUCCI, N.D.RYAN and J.BOWLES, Applications of Stainless Steels 92. M.Nordberg et al. eds. Jernkontoret, Stockholm (1992). p. 924.
19) H.J.McQUEEN, N.D.RYAN, E.EVANGELISTA and X.XIA, Proc. 34th Mechanical Working and Steel Processing. Iron and Steel Society, AIME. Warrendale (1993). p.101.
20) S.STRAUB and W. BLUM, Hot Workability of Steels and Light Alloys Composites. H.J.McQueen et al. eds. Met.Soc. CIM, Montreal (1996). p. 181.
21) H.J.McQUEEN, E.EVANGELISTA and N.D.RYAN, Stainless Steels 93. AIM, Milano (1993). vol.2, p. 289.
22) H.J.McQUEEN, N.D.RYAN and E.EVANGELISTA, Mat. Sci. Eng. A81 (1986). p.259.
23) E.EVANGELISTA, N.D.RYAN and H.J.McQUEEN, Met. Sci.Tech. 5 (1987). p.50.
24) N.D.RYANand H.J.McQUEEN, High Temp. Tech., 8 (1990). p.185.
25) H.J.McQUEEN, Y.CUI, B.LI, Q.MENG and N.D.RYAN, Strip Casting, Hot and Cold Working of Stainless Steels, Met.Soc.CIM, Montreal (1993). p. 181.
26) H.J.McQUEEN and N.D.RYAN, ICSMA9, Strength of Metals and Alloys, Brandon et al.eds. Freund Publishing House, London (1991). p.369.
27) H.J.McQUEEN, S.YUE, N.D.RYAN and E.FRY, Advanced Materials and Technologies, L.A.Dobrzanski ed. Silesian Tech. Univ. Gliwice (1995). p. 295.
28) H.J.McQUEEN, S.YUE, N.D.RYAN and E.FRY, J.Mat. Proc. Tech. 53 (1995). p. 293.
29) H.J.McQUEEN, O.OVERDAL, A.CINGARA and H.GJESTLAND, Mat.High Temp., 10 (1992) p.207.
30) H.J.McQUEEN, E.EVANGELISTA and N.D.RYAN, Recrystallization in Metals and Materials. T,Chandra ed. TMS-AIME Warrendale (1990). p. 89.
31) H.J.McQUEEN, J.BOWLES and N.D.RYAN, Microstruc. Sci., 19 (1989). p.357.
32) N.D.RYANand H.J.McQUEEN, New Developments in Stainless Steel Technology. R.A.Lula ed., ASM, Metals Park (1985). p.293.
33) N.D.RYAN and H.J.McQUEEN, J.Mech.Working Tech. 12 (1986). p. 79 and 323.
34) H.J.McQUEEN and C.IMBERT, Thermomechanical processing and Mechanical Properties of Hypereutectoid Steels, D.R.Lesuer et al.eds., TMS-AIME Warrendale (1997). p.55.
35) N.D.RYAN and H.J.McQUEEN, J.Mat.Proc.Tech., 36 (1993). p.103.
36) E.EVANGELISTA, H.J.McQUEEN and N.D.RYAN, Stainless Steels '91, ISIJ, Tokyo (1991). p.842.
37) J.W.SIMMONS, Mat.Sci.Eng. A297 (1996). p.159.
38) T.CHANDRA, O.P.DUNNE and P.CAMPBELL, Strength in Metals and Alloys (ICSMA7), H.J.McQueen et al. eds. Pergamon Press, Oxford (1985). p.947.
39) A.PINOL, D.IRIGUIEN, A.IZA, I.GUTIERREZ and J.URCOLA, Microstructural and crystallographic aspects of recrystallisation. N.Hansen et al. eds., Riso Natl. Laboratory, Roskilde, DK. (1995). p. 407.
40) W.BLUM and H.J.McQUEEN, ICAA5, Mat. Sci. Forum, 212-222 (1996). p. 31.
41) H.J.McQUEEN and W.BLUM, Recrystallization and related Topics, T.R.McNelley ed. Inst.Advanced Studies, Monterey (1997). p.123.
42) R.D.DOHERTY,....M.E.KASSNER,...H.J.McQUEEN, Sect. 9: Hot Worked State. Mat.Sci.Eng. 238 (1998). p.219.
43) H.J.McQUEEN and W.BLUM, ICAA 6, Aluminium Alloys, Physical and Mechanical Properties. T.Sato ed. Japan Inst.Met. Tokyo (1998). p. 99.
44) C.G..SCHMIDT, C.M.YOUNG, B.WALSER, R.H.KLUNDT and O.D.SHERBY, Metall.Trans. 13A (1982). p.447.
45) A. BELYAKOV, R. KAIBYSHEV and R.ZARIPOVA, Mater. Sci. Forum, 113-115 (1993). p.385.
46) A. BELYAKOV and R.KAIBYSHEV, Phys. Met. Metallov. 76 (1993). p.162.
47) T.MAKI, S.OKAGUCI and I.TAMURA, ICSMA 6, R.C.Gifkins ed. Pergamon Press, New York (1982). p.529.
48) P.CIZEK, R.RIMAN, V.SAFOK and V.CENY, Metallurgical Journal 43 (1989). p.99.
49) K.KATO, Y. SAITO, T. SAKAI, Trans. ISIJ 24 (1989). p. 94.
50) Y.MAEHARA, Trans. ISIJ 27 (1987). p.705.
51) Y.MAEHARA, Metall.Trans. 22A (1991). p.1083.

ABSTRACT

DEFORMAZIONE A CALDO DI ACCIAI INOSSIDABILI DUPLEX

Gli acciai inossidabili duplex, costituiti da isole di austenite in matrice ferritica, combinando le proprietà meccaniche e la resistenza alla corrosione degli acciai completamente ferritici o completamente austenitici rappresentano una classe di materiali strutturali ampiamente usati per la costruzione di sistemi per l'industria chimica, alimentare ed off-shore. Numerosi tipi sono disponibili sia per semilavorati che per getti, con composizione chimica riconducibile entro i seguenti limiti (% in peso): Cr: 22-25, Ni: 3-7, Mo: 3-4, N: 0,10-0,17. A differenza degli acciai inossidabili monofasici, che sono ampiamente studiati, pochi dati sono disponibili in bibliografia riguardanti la formabilità degli acciai duplex ad elevate temperature [5-11]. Nel presente lavoro vengono presentati risultati di ricerche eseguite nelle Università di Ancona e di Lovanio su diversi tipi di acciai duplex deformati mediante prove di torsione tra 1250-900°C e velocità di deformazione, $\dot{\varepsilon}$, tra 0.025-5.0 s^{-1}.

Nel lavoro vengono presi in esame la resistenza, la duttilità, la microstruttura e i meccanismi di ripristino degli acciai duplex 22.05 e 23.04. Questi aspetti sono importanti per determinare la formabilità degli acciai e quindi vengono usati per definire cicli di formatura e per costruire modelli sull'evoluzione strutturale.

La resistenza dà una indicazione delle forze necessarie per la formatura degli acciai. Essa dipende dalla temperatura, dalla velocità di deformazione e da fattori intrinseci del metallo come l'energia del difetto di ordinamento (stacking fault energy), il contenuto degli elementi in soluzione, la frazione in volume, composizione, grandezza e resistenza delle fasi secondarie. Le figure 1 e 2 mostrano l'andamento della tensione in funzione della deformazione e della temperatura a velocità di deformazione 1 s^{-1}; le curve presentano massimi di tensione seguiti da frattura o da un progressivo addolcimento (stato stazionario) che dipendono dalla temperatura e dalla composizione degli acciai. Ad $\dot{\varepsilon} = 1s^{-1}$ e a 1000°C il picco è seguito dalla frattura. La diminuzione della tensione di picco all'aumentare della temperatura è dovuta all'instaurarsi di cinetiche di ripristino e all'aumento della frazione della fase ferritica, fig. 3; questi fenomeni portano all'addolcimento degli acciai. N oltre ad esercitare benefici effetti sulla resistenza alla corrosione localizzata, stabilizza la fase austenitica anche ad elevate temperature producendo quindi un aumento della resistenza come mostrato nella figura 4.

La duttilità degli acciai testati alle diverse condizioni di temperatura e velocità di deformazione è un dato importante nei processi di formatura a caldo; nelle prove di torsione essa viene ricavata dalle curve tensione-deformazione come deformazione a rottura, ε_f. Nel 22.05 testato a T comprese tra 1000-1200°C e alle diverse condizioni riportate nella tabella 1, essendo l'austenite circa il 70% in volume, la duttilità aumenta con la temperatura; anche il 23.04 manifesta lo stesso comportamento anche se la fase austenitica diminuisce da 40 a 22%. La duttilità del 22.05 risulta superiore a quella del 23.04 in tutte le condizioni di prova.

La microstruttura rilevata con la microscopia ottica, mostra nella figura 6 l'austenite in forma di particelle irregolari di dimensioni comprese tra 20-150 µm. Dopo un lungo riscaldamento le particelle γ coalescono in forma sferica divenendo più piccole al crescere della T sopra 900°C. Nei campioni raffreddati dopo deformazione la fase α contiene sottograni che appaiono definiti più chiaramente come ε aumenta e più grandi al crescere della temperatura e al diminuire di $\dot{\varepsilon}$. Mantenendo i provini deformati alla temperatura di prova da 20 a 80 s prima del raffreddamento, si notano sottograni più grandi ed in alcune zone grani ricristallizzati staticamente nella fase α e grani ricristallizati staticamente in tutta la fase γ. La fase γ inoltre ricristallizza dinamicamente a deformazioni maggiori di quelle necessarie per li acciai austenitici alle stesse condizioni di T ed $\dot{\varepsilon}$. Questo effetto è riconducibile alla ripartizione della deformazione che avviene preferenzialmente nella fase α che è meno resistente della γ. I campioni osservati al microscopio elettronico in trasmissione mostrano le due fasi chiaramente distinguibili. La ferrite mostra sottograni grandi e poligonizzati anche dopo basse deformazioni ma ad elevate $\dot{\varepsilon}$, l'austenite sviluppa sottostrutture a maggiore densità di dislocazioni in celle più piccole rispetto a quelle della ferrite e mostra ricristallizzazione dinamica a tensioni critiche più elevate di quelle dei semplici acciai inossidabili austenitici dovuta all'assenza di siti di nucleazione all'interno dei confini di grano delle particelle γ.

La ricristallizzazione dinamica nella fase γ è la causa del picco nella curva tensione-deformazione; nella fase α tale ricristallizzazione non avviene. Nel corso della ricottura la ferrite manifesta notevole rinvenimento statico prima della ricristallizzazione mentre la fase γ ricristallizza facilmente in grani contenenti geminati.

Gli acciai austenitici invece avendo un limitato rinvenimento dinamico, a valori critici di deformazione ricristallizzano dinamicamente producendo un addolcimento notevole che nella curva tensione-deformazione è rappresentato da una caduta dei valori della tensione; il grafico presenta quindi un picco seguito da uno stato stazionario della tensione. I grani ricristallizzati si mantengono equiassici e la loro grandezza dipende dalla temperatura e dalla velocità di deformazione; tendono a crescere quando la temperatura aumenta e la velocità di deformazione diminuisce.