# DEFORMATION-INDUCED MARTENSITIC CHARACTERISITCS IN 304 STAINLESS STEEL DURING DEEP DRAWING

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***Abstract.*** *The effect of the deformation on the strain induced martensite ($\alpha'$) in an AISI 304 stainless steel which showed the Delayed Cracking Phenomenon on deep drawing operations was evaluated. The double forming of this metastable austenitic stainless steel (which was broken down into five stages) was performed with a 56mm initial blank diameter and 33 and 26mm punch diameters giving drawing ratios of 1.7 and 2.15, respectively. The last drawing ratio was determined as the Limiting Drawing Ratio of the Delayed Ccrackin, which was described as LDR-DC. Round samples of each stage have been cut to characterize the microstructure and determine the volume fraction of $\alpha'$martensite formed during the flow of the metal toward to the die. It was found that the amount of $\alpha'$martensite has increased with the strain in the deformation zone of the forming operations. The formed martensite has shown lath type morphology. The volume fraction of strain induced martensite has reached values close to 60% at the top edge of redrawn cups satisfying the condition previously stated by some authors for the occurrence of the phenomenon.*

***Keywords****: Delayed Cracking Phenomenon, strain induced martensite, deep drawing operation, austenitic stainless steel*

## 1. INTRODUCTION

The transformation from austenite to martensite in austenitic stainless steel accomplished by deformation of the parent austenite phase can result in martensite that can have either body-centered cubic (bcc) or hexagonal close-packed (hcp) crystal structures, $\alpha'$e $\varepsilon$ martensite, respectively [Lacombe, *et al.*, 1993]. The deformation induced martensite can be classified as stress assited or strain induced. Strain induced martensite is a direct consequence of plastic deformation and its production appears to be quite common in ferrous system. The martensite formed shows the lath type morphology.

It is well established that shear band intersections can be very effective nucleation sites for $\alpha'$martensite [Maxwell, 1974; Mangonon and Thomas, 1970; Lecroisey and Pineau, 1972; Olson and Cohen, 1975, Shrinivas *et al*., 1995]. The operative shear band can be in the form of deformation twins, stackings faults and $\varepsilon$ martensites [Raman and Padmanabhan, 1994]. The growth of the volume fraction of martensite can be attribuited to the increase in $\alpha'$ nucleating sites caused by increased strain [Shirinivas et al., 1995].

The deformation induced martensite brings improvement on mechanical strength and ductility, which is desirable for the high formability required by several forming processes. On the other hand, the martensitic transformation can have unfavorable effects, specially in the field of deep drawing in which the Delayed Cracking Phenomenon is prone to occurr.

The phenomenon is characterized by cracks opened at the top edge of cups deep drawn successfully. The occurrence of the phenomenon is related to the chemical composition of the austenitic stainless steel, martensitic transformation, residual stress and probably, to hydrogen content [Schaller *et al*., 1972; Wang and Gong, 2002; Sumitomo *et al*., 1981 and Hoshino, 1977].

In order to study the effect of the martensitic transformation on the Delayed Cracking Phenomenon, mainly the characteristics of $\alpha'$ formation, the deep drawing and redrawing operations were broken down into stages and microstructural analisys were carried out on the deformation zones of each one operation. In deep drawing, the deformation zone is placed at the flange area formed as the material flows into the die cavity. The stresses in the flange are compressive in the circunferential direction and tensile in the radial direction [Hosford, 2005]. The redrawing operation produces cups with a greater height-to-diameter ratio. The distribution of the stresses in the deformation zone are qualitatively equal to those occurring during the first drawn. Because of the generally conical shape of the die entrance an additional normal pressure, caused by the tangencial compressive stress, presses the cup against the die (Lange, 1985). So, the entire redrawn cup wall is forced to yield into the die cavity causing further strain.

There is a limit to the amount of reduction that can be achieved. It can be shown that for an isotropic material, the ratio of $d_b/d_p$ that the material can be drawn (Limiting Drawing Ratio or LDR) is

$$LDR = \frac{d_b}{d_p} \tag{1}$$

in which $d_b$ is the blank diameter and $d_p$ is the punch diameter [Hosford, 2005].

The purpose of this article was to report the microstructural variation during room temperature deep drawing and redrawing processes in an AISI 304 austenitic stainless steel that has shown the Delayed Cracking Phenomenon. It was observed the effect of strain during the double forming operation on the α´martensite characteristics and the volume fraction of this phase in the deformation zone of each process of forming.

## 2. EXPERIMENTAL DETAILS

### 2.1. Material

The chemical composition and $Md_{30}$ temperature of the 304 stainless steel (hereinafter labeled A) used in this study are shown in Tab. 1. The steel, which is a commercial grade of AISI 304, was received as a 0.6mm thickness sheet, previously subjected to an isothermal annealing for 30s at 1100 ºC. The $Md_{30}$ temperature of the A steel was measured according to the method used by Castro (2003) in her thesis.

Table 1 . Chemical Composition of AISI 304 austenitic stainless steel (in weight percent) and its $Md_{30}$ temperature (in degree Celsius).

| Steel | C | N | Si | Mn | Cr | Ni | Cu | Mo | Nb | $Md_{30}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| A | 0,0637 | 0,0373 | 0,382 | 1,329 | 18,27 | 8,03 | 0,06 | 0,039 | 0,006 | 4,0 |

The microstructure of the as-received steel is presented in Fig. 1. It is a single-phase polycristal (100% austenite) with equiaxed grains of about 19µm.

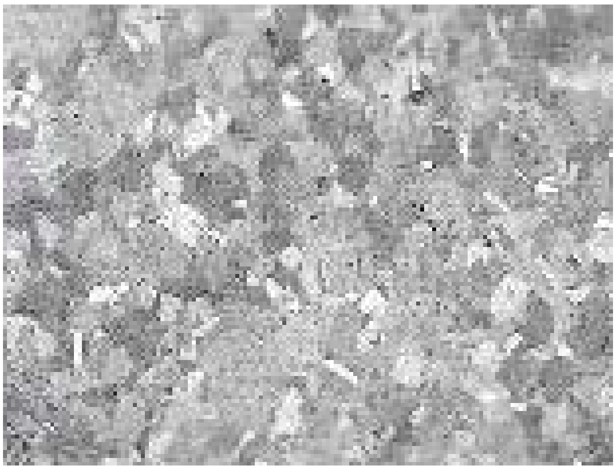


Figure 1. Microstructure of the as-received A austenitic stainless steel.

### 2.2. Deep Drawing Tests

The A steel was subjected to a double step drawing method by using an Erichsen 142-40 forming machine. Deep Drawing tests were carried out at room temperature with a 600mm/min deep drawing rate, at different drawing ratios (DR) by changing the various blank diameters (56, 60, 63, 65mm), using a 33mm punch diameter. The redrawn operations were performed with a 26mm punch diameter. In order to avoid wrinkle in the flange area, a blank holder was used which was pressed with pressures of 10-12kN against lubricated sheets of A steel. In the redrawing operation, the pressures have ranged of 4 to 6kN. The metal sheets were lubricated with molybdenum and PVC sheet.

One set of 15 drawn and redrawn cups were held at room temperature, at least of 24h, and periodically observed to evaluate the number of cracks opened in the cups edges. The Limiting drawing Ratio of Delayed Cracking Phenomenon

(LDR-DC) was defined as the minor drawing ratio for which, at least, one cup has splitted in cracks during a period of 24h after the final of the forming operation.

The deep drawn and the redrawn operations were broken down into stages in order to study the effect of strain on the formation of α´martensite. The stages were established at each 5mm of punch stroke, when the preforms were taken away from the press machine.

**2.3. Volume Fraction of Martensite Evaluation**

The martensite (α´) obtained from plastically deformed austenite (γ) in the deformation zone from the wall cups in each stage of the drawn and redrawn processes was measured, at the surface, by X-ray diffraction using CuKα radiation and a graphite crystal monochromator of a Philips PW70 Diffractometer. The X-ray diffraction and the microstructural analysis were carried out in small round specimens cut from the deformation zone of each deformation stage in a Metals Research Servomet Spark machine. The Servomet removes metal from the area in the immediate vicinity of the tool without mechanical contact with the work by generating a series of controlled spark discharges. Each spark removes a small crater of metal by melting and vaporization. The samples were etched with a HCl, $HNO_3$, acetic acid and glycerol solution to remove effects of cutting and surface impurities.

The semi-quantitative determination of volume fraction of phases by X-ray diffraction was based on the principle that the total integrated intensity of all diffraction peaks of each phase in a mixture is proportional to the volume fraction of that phase.

**2.4. Microstructural Analisys**

The samples used for X-ray diffraction were cold mounted in polyester resin in their transverse section, hand ground on SiC papers with grits of #320, #400, #600, #800, #900 and #1500, polished on diamond polishing pastes of 3 and 1 μm. The deformed layer due to the grinding and polishing was removed by eletropolishing in an electrolytic solution consisting of 50ml of percloric acid ($HClO_4$) and 950ml of methanol ($CH_3OH$), employing a d.c supply operated at 12V during 4s and. In sequence, the samples were rinsed in detergent and ethanol and dried with hot air.

The microstructure of the samples was revealed by etching them with an oxalic acid solution (10g oxalic acid, 100ml distilled water also using a d.c supply operated at 6V during 8s. A Leica optical Microscope was used to observe the microstructure and the images were taken with an image analysis software, Image Pro.

**3. RESULTS AND DISCUSSON**

**3.1 Limiting Drawing Ratio of Delayed Cracking (LDR-DC)**

The Delayed Cracking Phenomenon of the A austenitic stainless steel has occurred for the redrawn cup formed with the 56mm blank diameter when a crack of 1.0mm was observed at the cup edge in a period of 24h. This condition resulted in a drawing ratio DR=2.15. According to the previous definition, this drawing ratio was the Limiting Drawing Ratio of Delayed Cracking (LDR-DC). Figure 2 shows one redrawn cup of A austenitic stainless steel in which the phenomenon of Delayed Cracking has appeared.

Figure 2. Delayed Cracking Phenomenon in A stainless steel for LDR-DC=2.15.

### 3.3 Stages of the Double Forming Operation of A Austenitic Stainless Steel

Figures 3 and 4 show the stages in which the double forming operations were broken down for the A stainless steel in its blank of 56mm. Both the operations were broken into five stages (including the blank for deep drawing operation). The strains at deformation zone, measured by ASAME TARGET software, as well as the punch strokes used to step the forming operations, are presented in Tab. 2.

Figure 3. Stages of deep drawing operation.

Figure 4. Stages of redrawing operation.

Table 2. Deformations associated to each punch stroke during deep drawing and redrawing operations.

| Punch Stroke (mm) | Stage | True Strain (%) | |
|---|---|---|---|
| | | Deep Drawing | Redrawing |
| 0 | blank | 0 | 0 |
| 5 | 1 | 1,8 | 15,7 |
| 10 | 2 | 6,5 | 18,8 |
| 15 | 3 | 12,1 | 22,3 |
| 20 | 4 | 12,7 | 25,8 |
| 25 | 5 | - | 25,1 |

### 3.4 Volume Fraction of α´ Martensite Vs Deformation During Forming Operations

The variations in the volume fraction of α´ martensite with the deformation at each stage of deep drawn and redrawn operations are shown in Figs. 5 and 6, respectively. For both operations, the volume fraction of strain induced martensite increases with deformations during the forming. However, it must be noted that the amount of α´ martensite and the deformation associated to its formation are higher in the redrawn operation. The volume fraction of martensite close to 60% near the top of the redrawn cup is similar to that observed by Frehn and Bleck (2003) and Santos *et al.* (2005) in others grades of austenitic stainless steels in which the Delayed Cracking Phenomenon has appeared.

### 3.5 Microstructural Evolution During Room Temperature Deep Drawing and Redrawing Operations

On the first stages of deep drawing operation (deformation close to 7% strain) the only microstructural feature observed in the samples cut from deformation zone was the presence of the grain boundaries of the parent austenite phase. This fact is consistent with the X-ray diffraction profiles in which no α´martensite peak was observed. On increasing the deformation to about 12% strain, it was possible to observe the martensite nucleation at the grain boundaries. The martensite formed has had the typical morphology of lath type. Individual laths typically have narrow dimension. These features can be seen in Fig 7.

An example of microstructure obtained in the samples deformed over 12% strain is shown in Fig. 8. The figure shows blocks of lath martensite formed and some austenite.

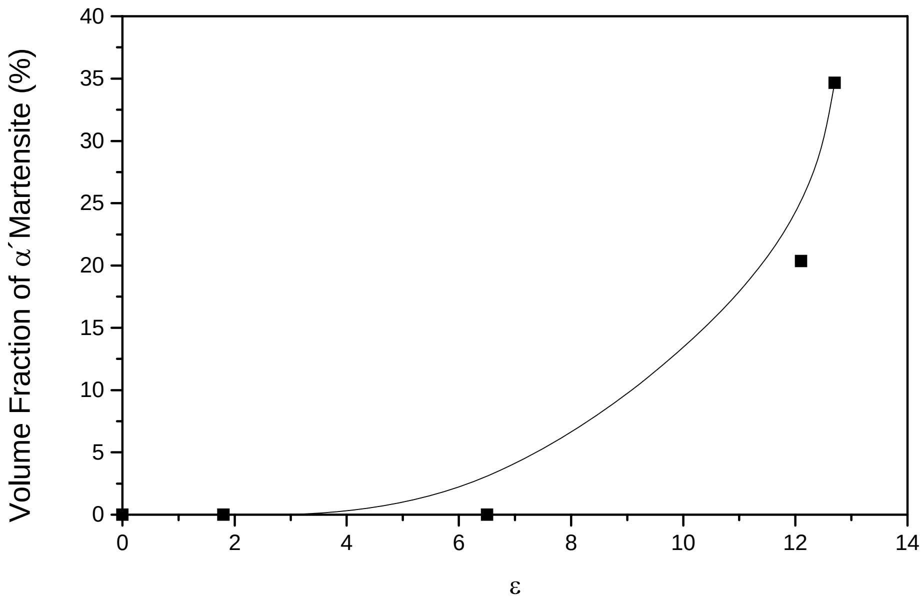



Figure 5. Variation in the percent volume fraction of α´ martensite as function of deformation during deep drawing operation in A stainless steel for LDR-DC.

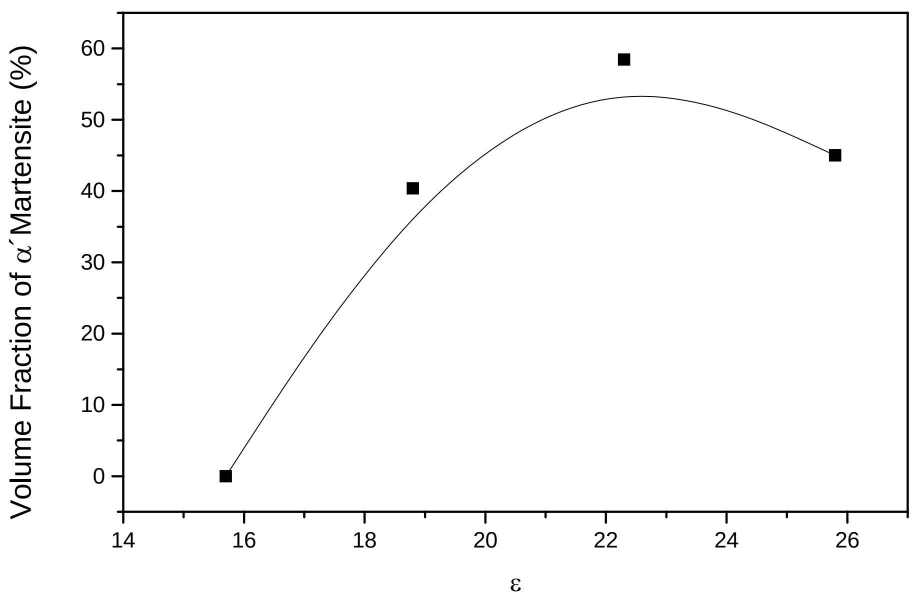



Figure 6. Variation in the percent volume fraction of α´ martensite as function of deformation during redrawing operation in A stainless steel for LDR-DC.

Figure 9 shows the microstructure developed after A stainless steel samples have been deformed to strains over 16% during redrawing operation. These figures clearly show the increase in α´content probably attributed to the increase in α´ nucleating centers caused by increased strain [Shrinivas *et al*., 1995]. Once again, as in Fig. 7, typical martensite laths formed into austenite grain can be seen. At the final of the redrawing operation, blocks of lath martensite and the parent austenite were present.

It may be speculated that the growth of α´martensite has occurred due to the increase in strain during the forming operation [Shrinivas *et al*., 1995]. According to Shrinivas *et al*., (1995) the number of shear bands intersections, which has been found to be the main nucleation site for α´martensite formation during rolling of an AISI 304 austenitic stainless steel, was dependent of stacking fault energy (SFE) value. The lower SFE value for 304 stainless steel, 21mJ/m$^2$ [Murr, 1969], resulted in more planar slips, indicating a larger number of shear band intersections and sites for martensite nucleation.

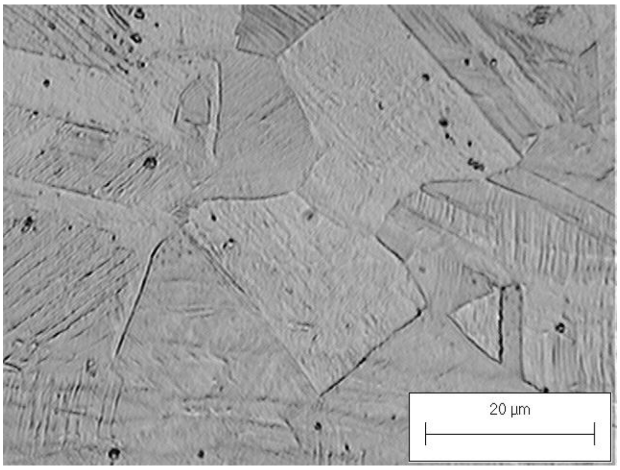



Figure 7. Optical micrographs of samples presenting nucleation of α´martensite in A steel during room temperature deep drawing at 12% strain level.

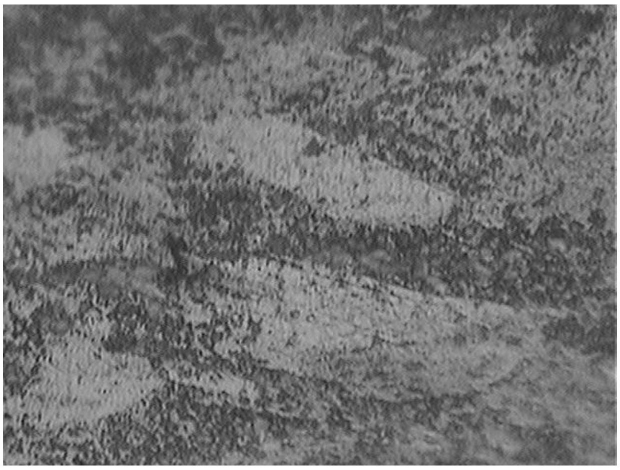
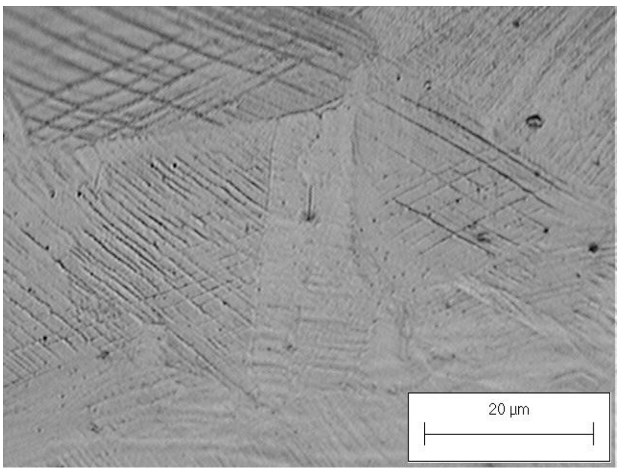
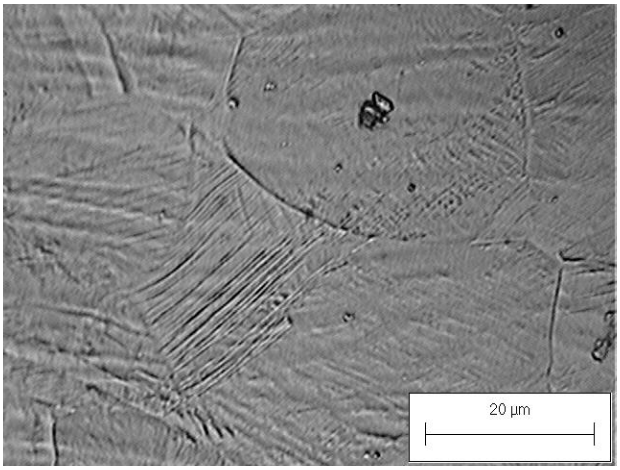


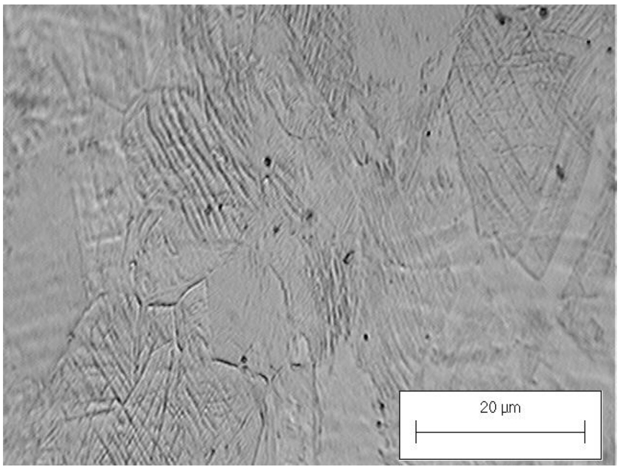
Figure 8. Optical micrographs of samples showing blocks of α´martensite formed at high levels of strains in deep drawing operation.




(a)




(b)




(c)

Figure 9.Optical micrographs of samples deformed in redrawing operation to 19% strain (a), 22% strain (b) and 26% strain levels.

## 3. CONCLUSIONS

The volume fraction of α´martensite formed during deep drawing and redrawing operations increases as a result of increase in strain during the forming operation. This fact must be related to the increase in number of shear band intersections, which could be the main nucleation site for α´martensite.

The lath type morphology of α´martensite has been found in A austenitic stainless steel for any strain level.

The martensite content in deformation zone of the drawing and redrawing operations increased gradually from the bottom to the height of the cups at each stage.

The Delayed Cracking Phenomenon was associated with a high amount of α' martensite, close to 60% in the top of the cups.

## 3. ACKNOWLEDGEMENTS

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## 4. REFERENCES

Castro, C.S.B., "Efeito da Adição de Cobre nas Propriedades Mecânicas de Aços inoxidáveis Austeníticos" . Belo Horizonte: Escola de Engenharia da UFMG, 2003. 154p (Tese, Doutorado em Engenharia Metalúrgica e de Minas).

Frehn, A., Bleck, W., Effect of Austenite Stability on the Mechanical Properties and Delayed Cracking in Metastable Austenitic Steels, *Stainless Steel World (The Netherlands)*, v. 15, no. 1, pp. 40-45 2003.

Hosford, W.F., Mechanical Behavior of Materials, Cambridge University Press., 1st edition, New York, 2005

Hoshino, K.; Effect of Chemical Composition on Season Cracking of Metastable Stainless Steel. *Transactions ISIJ*, v. 20, pp. 147-153, 1977.

Lacombe, P., Baroux, B., Beranger, G.; Stainless Steel, Les Editions de Physique, first edition, Les Ulis, 1993.

Lacroisey, F., Pineau, A., Martensitic Transformations Induced by Plastic Deformation in the Fe-Ni-Cr-C System, *Metallurgical Transactions*, v. 3, pp. 387-396, 1972.

Lange, K., *Handbook of Metal Forming*. McGraw-Hill Book Company, first edition, New York. 1985.

Mangonon, JR.P.L., Thomas G., The Martensite Phases in 304 Stainless Steel, *Metallurgical Transactions A*, v. 1, pp. 1577-1586, 1970.

Maxwell, P.G., Goldberg, A., Shyne, J.C., *Metallurgical Transactions*, v. 5, pp. 1305-1318, 1974.

Olson, G.B., Cohen, M., Metallurgical Transactions A, v. 7A, p. 1897, 1915.

Raman, S.G. S., Padmanabhan, K.A., Tensile deformation-induced martensitic transformation in AISI 304L Austenitic Stainless Steel, *Journal of Materials Science Letters*, v. 13, pp. 389-392, 1994.

Santos, M. R., Alves, H.J.B., Lopes, R.G, Gonzalez, B.M., "Effect of Copper Addition on Delayed Cracking Phenomenon of Deep Drawn- and Redrawn Cups of Austenitic Stainless Steels" **,** Proceedings of the 17th International Congress of Mechanical Engineering. Ouro Preto, Brazil, november 6-11, 2005.

Schaller, F.W., Schimid, T.E., Snape, E., Delayed Cracking of Deep Drawn Stainless Steel, *Sheet Metal Industries*, v.10, pp.621-624, 1972.

Shrinivas, V., Varma, S.K., Murr, L.E., Deformation-Induced Martensitic Characteristics in 304 and 316 Stainless Steels During Room-Temperature Rolling, *Metallurgical and Materials Transactions A*, v.26A, pp. 661-671, 1995.

Sumitomo, H., Sawatani, T., Arakawa, M., Effect of Chemical Compositions on Delayed Cracking of Deep Drawn Cup of 17%Cr-7%Ni-2%Cu Steel, *ISIJ Meeting*, p. 102, 1981.

Wang, Z., Gong, B. Residual Stress in the Forming of Materials. *ASM International Handbook of Residual Stress and Deformation of Steel* (USA), pp. 141-149, 2002.

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