



EVALUATION OF MECHANICAL PROPERTIES IN OF DOUBLE-DIP GALVANIZED COATINGS ON CARBON STEEL

EVALUACIÓN DE PROPIEDADES MECÁNICAS EN RECUBRIMIENTOS GALVANIZADOS POR DOBLE INMERSIÓN EN CALIENTE SOBRE ACERO AL CARBONO

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Resumen

Poco se conoce sobre las condiciones operacionales, la microestructura y propiedades de los recubrimientos fabricados por doble inmersión en caliente. Este trabajo tiene como objetivo evaluar propiedades mecánicas de recubrimientos Zn/Zn-5%Al aplicados por la técnica de doble inmersión en caliente, variando los tiempos de inmersión en los baños líquidos. Para la evaluación se realizaron perfiles de microdureza Vickers y ensayos de doblez. Los perfiles de microdureza para diferentes tiempos de inmersión presentan similitudes, mostrando gran heterogeneidad debido a las características microestructurales. Se observa que al aumentar el tiempo de inmersión disminuye el ángulo crítico y el tiempo de inmersión no influye significativamente en la densidad de grietas confinadas y no confinadas. Se concluye que la ductilidad de los recubrimientos se ve influenciada por el espesor total de los mismos, y posiblemente por el espesor de las diferentes zonas y esfuerzos residuales, siendo las muestras recubiertas con tiempo de inmersión de 60 segundos, las que presentan mejor comportamiento ante el ensayo de doblez.

Palabras clave: microgrietas, doble inmersión, recubrimientos galvanizados.

Abstract

Little is known about the operational conditions, the microstructure and properties of the coatings manufactured by hot double-dip. The objective of this work is to evaluate the mechanical properties of Zn / Zn-5%Al coatings applied by the hot double-dip technique, varying the immersion times in liquid baths. For the evaluation, Vickers microhardness profiles and bending tests were made. The microhardness profiles for different immersion times show similarities, exhibiting great heterogeneity due to the microstructural characteristics. It is observed that increasing the immersion time decreases the critical angle, and the immersion time does not significantly influence the density of confined and unconfined cracks. It is concluded that the ductility of the coatings is influenced by their total thickness, and possibly by the thickness of the different areas and residual stresses, with the samples being coated for a 60 s immersion time, which present better behavior in the bending test.

Keywords: Microcrack, double-dip, galvanized coatings.

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1. Introduction

After processes of hot galvanizing, the pieces of coated steel may be subjected to plastic deformations in pressing, stamped and bending processes. These processes cause a great deformation in the structure of steels, which in turn may induce the beginning and propagation of cracks in the coatings. Once the cracks propagate, their openings provide air and humidity passages that lead to adverse oxidation reactions and corrosion, both in the coatings and in the steel substrates. The mechanical behavior of galvanized coatings on steels, may alter the performance of its response to operations that involve plastic deformations.

While the behavior regarding corrosion of galvanized steels has been rigorously investigated, the mechanical behavior of the galvanized coatings is currently limited [1].

In general, the hot galvanized coatings are complex multilayer systems, constituted by phases or layers with different thermomechanical properties. This makes difficult to analyze the mechanical behavior of the system steel/coating; in addition, the lack of information about the thermomechanical properties of the individual phases that constitute the coating should be added to this difficulty, as well as the properties of the interfaces [2].

The failures of hot galvanized coatings have been related with the residual stresses generated in their manufacturing process. The microcracks induced in the solidification process frequently occur on the galvanized coating, due to large mismatch between the thermal expansion coefficients of the zinc coating and the steel substrate. This may significantly influence on the density of cracks that are formed in the zinc layer, and in the further delamination of the coating under load [3].

There is a diversity of mechanical properties that may be evaluated in the coatings. Both the elastic and plastic properties are important for a specific application or demand. The ductility of the coatings depends on factors such as grain size, crystallographic orientation, working temperature, thickness of the coating, chemical composition, morphology and distribution of the phases that constitute the microstructure of the coating [4].

The double-dip process consists of consecutively submersing the steel in two liquid baths with different chemical composition. It is important to mention that most of the galvanizing processes by hot immersion are of simple or unique immersion, where the steel is submersed in a bath with specific chemical composition that provides the coating its mechanical, chemical and physical properties.

Nevertheless, the operational conditions in the manufacturing process, the microstructure and properties of the Zn/Zn-5%Al double-dip galvanized coat-

ings have been scarcely studied. It is known that Galfan®(Zn-5%Al) baths provide greater resistance to corrosion and better ductility than conventional Zn baths. These characteristics may be found in the external zone of the coating, once the second immersion has been carried out, without requiring to change the fluxing systems in the preparation of the steel. Information regarding the possible industrial application of double-dip coatings has been little disseminated, but it is estimated that they can be used in components located in more severe corrosion environments, where traditional Zn coatings exhibit a lower protecting performance.

For that matter, the objective of this research work is to evaluate the mechanical properties of Zn/Zn-5%Al coatings applied using the hot double-dip technique, with varying times of immersion in the liquid baths.

2. Materials and methods

Samples of AISI 1020 steel, of dimensions 100 mm x 38 mm x 3 mm, were employed to conduct this research. The surfaces of the samples were degreased with 17 % NaOH for 5 minutes at 60 °C; for the abrasion they were submersed in a solution of 18% hydrochloric acid for 1.5 minutes at 80 °C and, at last, they were submersed for 5 minutes in a 30 g/600 ml solution of ammonia chloride for fluxing, at a temperature of 70 °C, and then dried with air at ambient temperature. The double-dip hot galvanizing process was carried out experimentally, in a vertical electrical furnace that contains two crucible with each of the liquid baths, namely a type I bath of pure Zn, and a type II bath of Zn with 5 % in weight of Al (Galfan®); the temperature of the baths was 550 °C ± 10 °C.

The working temperature was determined by means of previous tests, where it was found that the fluidity of the dip baths was notably low at smaller temperatures, thus making difficult the immersion and emersion of the steel samples in the liquid baths.

It is important to remark, that the microstructural evaluation of the double-dip coating under the same operational conditions was previously reported [5]; the microstructural characteristics described in [5] will be taken as reference in this work.

The Zn/Zn-5%Al coatings were made with different immersion times: 30 s in each bath (pure Zn and Zn with 5% in weight of Al), for a total immersion time of 60 s; 45 s in each bath for a total immersion time of 90 s and 60 s in each bath for a total immersion time of 120 s; 3 samples were galvanized with each total immersion time. Table 1 shows the operational parameters of the double-dip galvanized process.

The transverse sections of the double-dip galvanized samples were prepared using conventional methods, cut with abrasive disc, roughing with sandpaper,

and mechanical polished with alumina suspension, to determine the thicknesses of the coatings by means of optical microscopy and carry out Vickers microhardness profiles.

Table 1. Parameters of the double-dip galvanized process

Operational parameters	
Chemical composition of the baths	Type I bath: 100 % Zn Type II bath: Zn-5 % weight Al
Time of immersion in each bath	30, 45 y 60 s
Total immersion time	60, 90 y 120 s
Immersion way	Quieto
Temperature of the baths	550 °C ± 10 °C
Cooling after the extraction	Quiet air

The Vickers microhardness tests were conducted taking measurements from the steel/coating interface through the coating up to its surface, with a load of 50 g. Six microhardness profiles were carried out for each total immersion time, making indentations every 50 µm (approximately), and the obtained values were plotted vs. distance, for each total immersion time.

In order to evaluate the relative ductility of the coatings, the samples were deformed up to the critical angle, which is the angle under which the beginning of the macroscopic cracking in the critical deformation zone, can be visually observed at the moment of the test [6]. The B arrangement for semi-guided tests, suggested by the standard ASTM E-290 [7], was utilized for the bending test (see Figure 1).

Afterwards, the transverse sections of the samples, tested by means of optical microscopy, were examined, in order to identify the different types of cracks, and describe qualitative and quantitatively the damage induced by the flexion. For this purpose, it was determined the density of cracks (number of cracks/mm) formed perpendicularly to the steel/coating interface, in the tensioned zone of the samples. These measurements were carried out along a 20 mm long arc, symmetric with respect to the maximum point of flexion (Zone A), as shown in Figure 1 [8].

On the other hand, a statistical analysis of the results was carried out by means of a unidirectional variance analysis (ANOVA), comparing the probability factors obtained with the Fisher F statistic, for a reliability of 95 %. The total immersion times were related with the thickness of the coating, the critical angle and the density of confined and not confined cracks

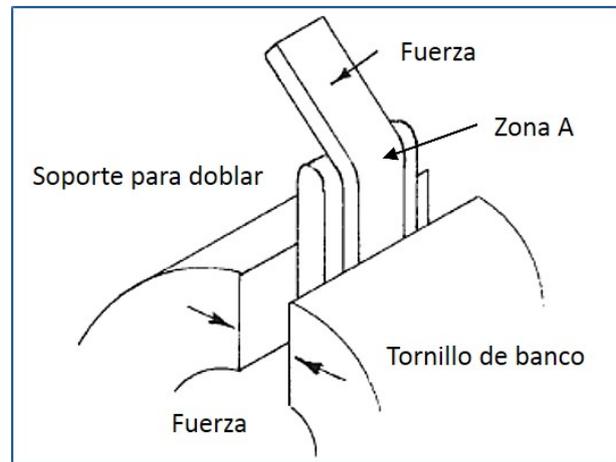


Figure 1. Scheme of the flexion test according to the ASTM E-290 standard. Arrangement B for semi-guided bending test of thin samples with a retained extreme; zone A will be the examined zone [7].

3. Results and discussion

All the coatings obtained using the double-dip method, are according the specifications of the ASTM A-123 standard: «Standard Specification for Zinc (Hot-Dip Galvanized) Coatings on Iron and Steel Products» [9] (see Figure 2).

The coatings presented commercially acceptable superficial characteristics. Regarding the superficial finish, all samples exhibit continuity and do not have zones without coating nor of varying roughness. With respect to the superficial appearance, the double-dip galvanized coatings do not have ampoules nor slag.

The thicknesses of the coatings were sufficiently large, in the range 450-650 µm (see Figure 3), compared to commercial zinc coatings that are in the order of 100 µm. Figure 3 clearly shows that the thicknesses of the coatings vary significantly with the total immersion time: as the total immersion time increases, the total thickness of the coating also increases.



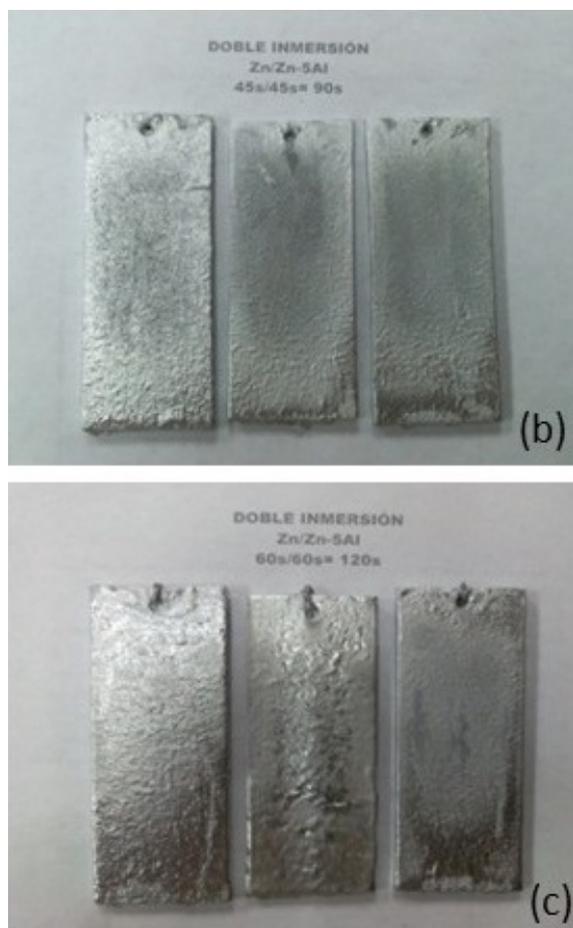


Figure 2. Muestras de acero recubiertas con la técnica de doble inmersión, para diferentes tiempos de inmersión a) 60 s, b) 90 s y c) 120 s.

The ANOVA carried out shows that $F_{experimental} = 171,51$ and $F_{0,05}(2,51) = 3,18$; i.e., $F_{experimental} > F_{0,05}(2,51)$ and $P_{experimental} < 0,05$; therefore, the null hypothesis is rejected and the thickness of the coating varies significantly with the total immersion time.

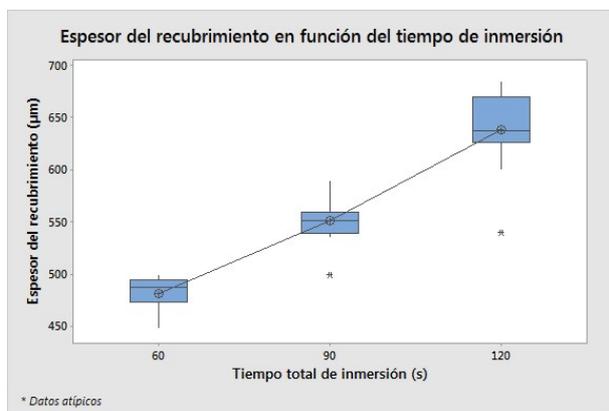


Figure 3. Gráfica de caja del ANOVA para el espesor total de los recubrimientos galvanizados por doble inmersión en función del tiempo total de inmersión.

The large thicknesses obtained, compared to commercial zinc coatings, suggest that the immersion in the second bath (Zn-5%Al) and the reactivity or synergy of both dip baths, determine the increase in the total thickness of the coating.

The reactivity of the chemical species, mainly Zn, Al and Fe and the growth kinetics of the formed phases in the second immersion, may be preponderant in the increase of the thickness of the double-dip galvanized coatings. Another important factor that may increase the velocity of the reactions and the growth kinetics is the working temperature, which was 550 °C; this value is utilized in the called galvanized at «high temperatures».

For the case of the double-dip galvanized coatings, it is difficult to determine which mechanism controls the kinetics of the total growth of the coating. It is estimated that for the first immersion bath (pure Zn), the growth of the coating thickness follows a nonlinear behavior with respect to the immersion time, as indicated by the literature [2, 10, 11]; but when the steel is immersed in the second bath, which contains 5 % of weight of aluminum, it could give place to the fast formation of Fe-Al-Zn compounds, which influences the type of behavior of the growth of the coating [5], thus increasing the velocity of initial growing, as shown by the large thicknesses obtained.

Nevertheless, the type of kinetics of the growth of the double-dip galvanized coating is still nonlinear, which indicates that the total mechanism that controls the growth of the coating is the diffusion of species, despite of the chemical reactions that can be generated in the second dip bath. It is estimated that the velocity at which these reactions occur, may determine the growth of the thickness of the coating in the second dip bath, but does not determine the type of kinetics of this growth.

The general microstructural characteristics independent of the total immersion time, were described in [5]. They defined three zones in the double-dip galvanized coatings: Zone I, constituted by phase δ of faceted morphology in the steel/coating interface, which significantly varies with the total immersion time and phase η ; zone II shows high microstructural heterogeneity and is mainly constituted by three phases, namely phase η which appears as a matrix, phase δ and Fe-Al-Zn micro-segregated ternary compounds of rounded morphology; and zone III is constituted by phases η and $Fe_2Al_5Zn_x$ compounds of rounded morphology.

It is evident in Figure 4 that there exists a similar trend in the microhardness values obtained for the three total immersion times. In zone I, specifically in the area adjacent to the steel/coating interface, the microhardnesses are elevated, with a general average of 254 HV, because it corresponds to the value of microhardness of phase δ ($FeZn_{10}Al_x - FeZn_7Al_x$).

After this area, a considerable decrease in the values

of microhardness is observed, with a general average of 19 HV, which corresponds to phase eta, η (pure Zn).

In zone II there exists a slight trend of the microhardness to increase to values in the range 100-200 HV, due to the presence of Fe-Al-Zn precipitated ternaries in a hard phase δ ; however, this zone presents a large microstructural heterogeneity and, as a consequence, there is a high variability of the values of microhardness. At last, in zone III the microhardness oscillates around 100 HV, in a microstructure basically constituted by $\text{Fe}_2\text{Al}_5\text{Zn}_x$ precipitated in a matrix η of practically pure Zn.

The values of microhardness for phase δ and η formed in zone I of the coatings, for each total immersion time, are similar to the values of microhardness reported by other authors [4, 10–14]. For zones II and III, it is difficult to find reference values of microhardness, especially for zone II because of the presence of great heterogeneity; the microhardness values of zone III could be compared with the hardness values in Galfan@coatings, but these values may vary depending on the cooling conditions of the coating after it is extracted from the bath, since it may modify its eutectic structure making it thinner for faster cooling. It has been found that for coatings with 4.5 % weight of Al, the microhardnesses are between 75.1 and 76.2 HV [15], slightly smaller than the obtained for zone III in this study, which are close to 100 HV.

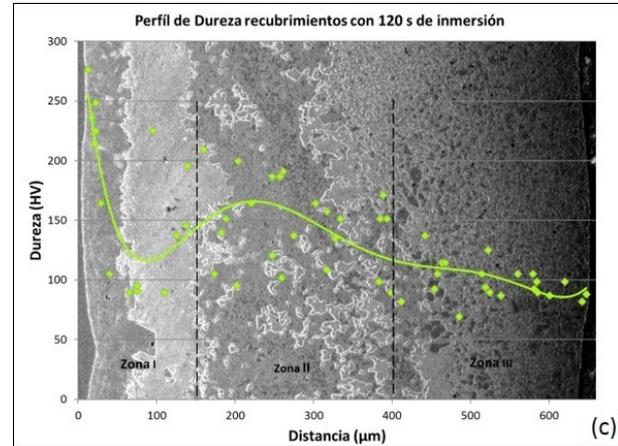
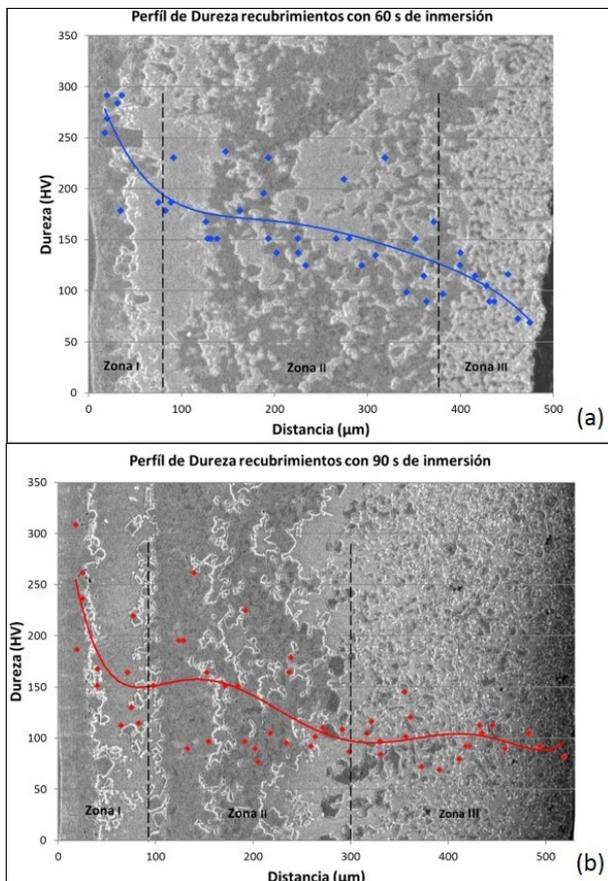


Figure 4. Vickers microhardness profiles for double-dip galvanized coatings, with total immersion time: a) 60 s, b) 90 s y c) 120 s.

The thickness of each zone of the double-dip galvanized coatings, depends on the immersion time in each bath; therefore, the trend of the microhardness values for each zone of the coatings is widened or reduced depending on the length of each zone. For example: the length of zone III in the coatings with an immersion time of 60s is much smaller compared to the length of zone III in the coatings with an immersion time of 120s, so the extension or trend of the microhardness values in this zone is broader in the coatings with 120s immersion time. This condition might influence the mechanical behavior of the coatings, specifically in their ductility.

Figure 5 shows the coated samples tested by bending up to the critical angle, for a total immersion time of 60s.

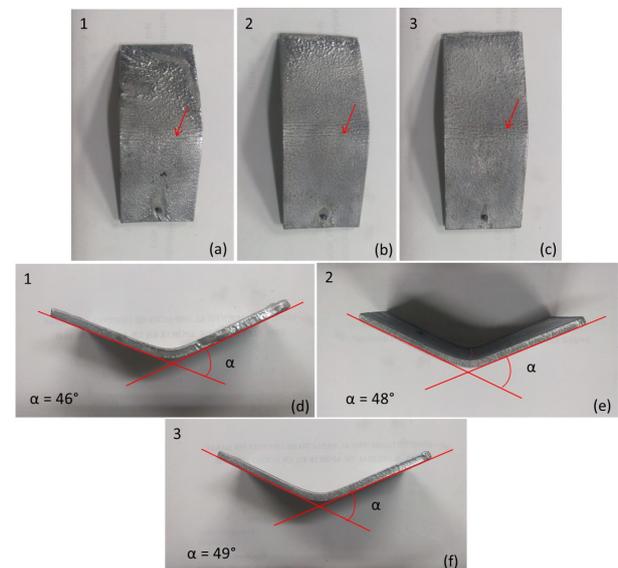


Figure 5. Samples 1, 2 y 3 with double-dip galvanized coatings with a total immersion time of 60s, tested by bending. The critical angle can be observed: superior view (a, b y c), transverse view (d, e y f).

The superior view of the surfaces of maximum bending in the tested coated samples is shown, where it can be seen the beginning of the macroscopic cracking of the coating (indicated by red arrows); the transverse view of the critical angle for each of the samples is also shown.

A unifactorial ANOVA variance analysis was conducted, to determine the influence of the total immersion time on the critical angle. For this experiment, the null hypothesis was that the critical angle does not vary with the total immersion time for the three levels under study, i.e. 60, 90 and 120 seconds.

Figure 6 is a box plot that shows the results of the ANOVA, and the influence of the total immersion time on the critical angle of the double-dip galvanized coatings. Observe the means and the intervals according to the standard deviation of the measured values of critical angle for each total immersion time. Since the mean values are significantly different, the representative boxes of each level of study (60, 90 y 120 s) do not overlap.

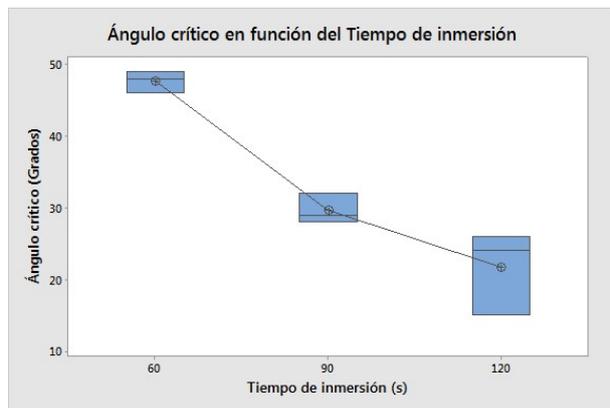


Figure 6. ANOVA box plot of the critical angle as a function of the total immersion time, in double-dip galvanized coatings.

The ANOVA shows that $F_{experimental} = 38,93$ and $F_{0,05}(2,6) = 5,14$; therefore, $F_{experimental} > F_{0,05}(2,6)$ and $P_{experimental} < 0,05$; therefore, the null hypothesis is rejected and the critical angle varies significantly with the total immersion time: as the total immersion time increases, the critical angle decreases.

In thick coatings, as the ones under study, the magnitude of the residual stress depends on the thickness of the coating. This residual stress develops during the formation of the coating and the further cooling; in general, it is produced during the formation of individual layers, due to the differences in the molar volume of each of these layers, as well as during the cooling from the immersion temperature, due to the mismatch of the thermal tensions as a result of the differences in the thermal expansion coefficient of the substrate and the different phases that constitute the coating; these

residual stresses significantly increase as the thickness of the coating increases [2, 16].

Due to this, it is expected that the critical angle varies with the total thickness of the coating. For large thicknesses, it is estimated that the residual stress will be greater in the coatings, and therefore pre-existing microcracks in phase δ , new microcracks formed in the same phase during the deformation, and in other zones of the coating subject to tension in the bending state, evolve to macrocracks which are visible at smaller bending angles, when compared with coatings of lesser thicknesses.

In bending tests, the critical angle has been related to the total thickness of the layer $\delta + \zeta$, phases adjacent to the steel/coating interface for traditional galvanized [6], determining that the critical angle decreases as the thicknesses of the intermetallic layers Zn-Fe increase. Nevertheless, for double-dip galvanized coatings, it is estimated that the critical bending angle depends, not only on the thickness of the phase δ but, as it has been already commented, also the total thickness of the coating has a significant influence due to the residual stresses generated in thick coatings. Another important factor that could have influence in the critical bending angle, are the thicknesses and sizes of the different zones of the double-dip galvanized coatings, and thus the mechanism of formation and growth of the microcracks generated in each of them.

The quantified cracks were classified in two types: not confined, which extend along the coating and expose the steel substrate to the atmosphere, and confined, which do not extend along the whole thickness of the coatings. The latter were sub-classified in a qualitative manner in the double-dip galvanized coatings.

Figure 7 is a box plot which shows the results of the ANOVA, and the influence of the total immersion time on the density of confined and not confined cracks of the double-dip galvanized coatings.

Similarly, observe the means and the intervals according to the standard deviation, of the measured values of crack densities for each total immersion time. Since the means are not significantly different, the representative boxes of each level under study (60, 90 y 120 s) overlap, which indicate that changes in the immersion times do not significantly influence the density of both types of crack.

The ANOVA shows that $F_{experimental} = 3,59$ for the density of confined cracks, the $F_{experimental} = 3,73$ for the density of not confined cracks and $F_{0,05}(2,6) = 5,14$; therefore, for both cases $F_{experimental} < F_{0,05}(2,6)$ y $P_{experimental} > 0,05$. Then, the null hypothesis is accepted, and the density of confined and not confined cracks do not vary significantly with the total immersion time, for double-dip galvanized coatings tested up to the critical angle.

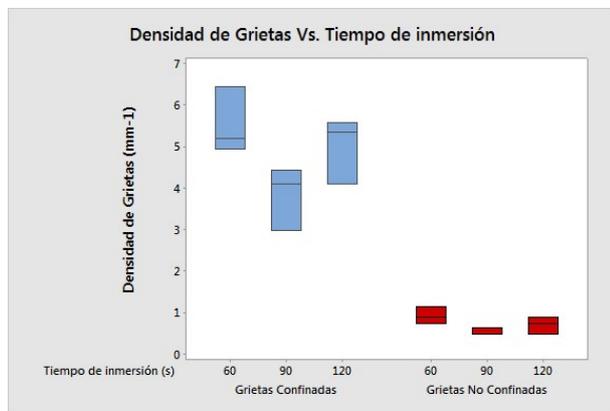


Figure 7. ANOVA box plot of the density of confined and not confined cracks as a function of the total immersion time, in double-dip galvanized coatings

Nevertheless, it is important to remark that the bending test applied to the double-dip coated samples, for the different total immersion times, was conducted up to the critical angle which, as has been statistically demonstrated, varies significantly with the total immersion time. Therefore, the samples were tested at different angles, for the different immersion times, up to the beginning of the macroscopic cracking in the critical deformation zone (see Figure 5).

This could explain why the density of the cracks do not vary significantly with the total immersion time. All the double-dip coated samples were tested by bending up to the macroscopic cracking and, hence have, in average, the same density of confined and not confined cracks.

In the double-dip galvanized coatings tested by bending up to the critical angle, independently of the total immersion time the following general characteristics and type of perpendicular microcracks to the steel substrate were observed, and studied qualitatively:

i. Confined cracks in phase δ , which could be in turn divided in microcracks pre-existing at the bending test, and microcracks formed in the bending test (Figure 8a); these represent the majority of confined cracks.

ii. Confined cracks that are present along the whole zone II of the coating, formed at the bending test (see Figure 8a).

iii. Confined cracks that extend from phase δ up to the end of zone II of the coatings. It is possible that these cracks result from the advancement of type i cracks in the bending test (see Figure 8a).

iv. Confined cracks that extend from the surface of zone III of the coatings up to zone II, formed during the bending test (see Figure 8b).

v. Not confined cracks that extend along the whole double-dip galvanized coating (see Figure 8b).

The macroscopic cracking of the coatings constitute the step previous to the failure, in general by delamination of the galvanized coatings; however, in the double-dip coated samples tested by bending up to the critical angle, there was no evidence of macroscopic delamination of the coating. The beginning of cracking longitudinal or parallel to the substrate in the steel/coating interface, was determined in the base of some not confined cracks.

It is important to remark that it was not noted longitudinal cracking inside the described zones or between them in any of the double-dip coated samples, which implies an excellent cohesion between the zones formed in each of the dip baths.

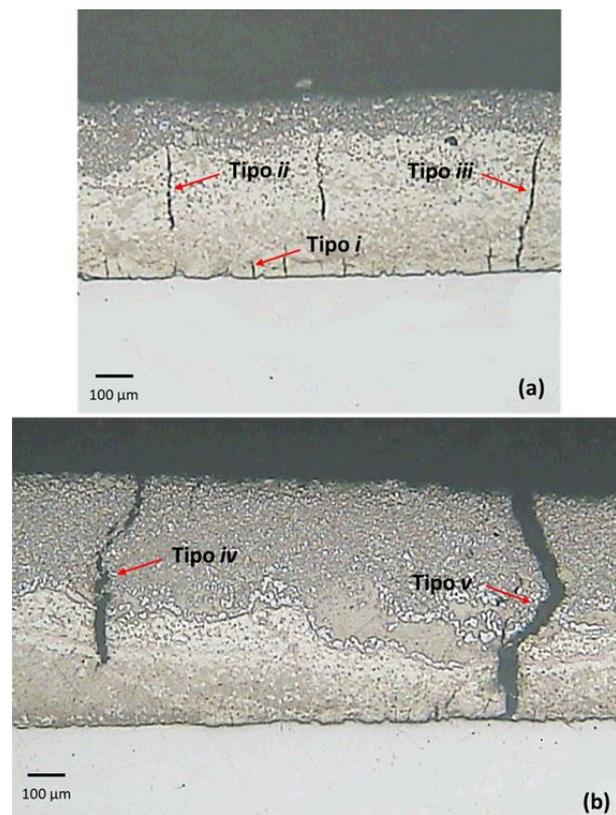


Figure 8. Optical micrography of the types of microcrack found in double-dip galvanized coatings tested up to the critical angle: a) Coating with a total immersion time of 60 s, showing microcracks of type i, ii and iii, b) Coating with a total immersion time of 120 s, showing microcracks of type iv and v.

Even though in the present study no observations were performed about the behavior in the initiation and propagation of cracks for different bending angles, based on what was observed in the double-dip galvanized coatings tested up to the critical angle, the initiation and propagation of cracks can be described in the following manner:

First, once the steel samples have been coated by the double-dip, there is a great quantity of residual stresses in the coating that increase with the total

thickness of the coatings, and cause the formation of confined microcracks in the phase δ of zone I, type i microcracks pre-existing at the bending test, which in general are perpendicular to the steel substrate, for a bending angle of 0° , α_0 .

Once the deformation of the double-dip coated samples begins in the bending test, new type i microcracks are generated in the phase δ , and it is possible that microcracks extending along the whole zone II will simultaneously start to generate, type ii microcracks for bending angles $\alpha_0 > \alpha_0$. It is important to remember that phase δ is one of the hardest in double-dip coatings, with an average microhardness of 254 HV. Zone II presents values of microhardness in the range 100-200 HV, with great microstructural heterogeneity; it is constituted by phases δ , η and Fe-Al-Zn ternary precipitates. The latter could act as stress concentrators that facilitate the growth and propagation of cracks, which would imply the presence of critical areas for the formation of new type ii microcracks.

The advancement of pre-existing cracks and the formed in the bending test, in phase δ towards zone II of the double-dip galvanized coating, require that these microcracks cross phase η in zone I. This phase is soft and the microcracks could tend to be blocked in it, without advancing towards zone II of the coatings; in fact, the majority of these cracks seem to stay as type i confined cracks, and represent the majority of the confined microcracks observed in the double-dip coatings; this could explain the difference between the density of confined and not confined cracks (Figure 7). Perhaps, only a small amount of these type i cracks will exceed the necessary critical stress to advance through phase η , and once in zone II (zone with more hardness than phase δ) continue advancing through its end, becoming type iii cracks for bending angles $\alpha_2 > \alpha_1$.

On the other hand, in the opposite direction, from the surface of the coating in zone III, even though this zone presents a microhardness ≈ 100 HV, with a soft matrix of η and $\text{Fe}_2\text{Al}_5\text{Zn}_x$ hard precipitates, microcracks resulting from the tensions generated by the bending test initiate and propagate towards the steel substrate; this zone is subjected to the greatest tension during the test, and type iv cracks are generated; these cracks formed on the surface penetrate towards the steel/coating interface, for bending angles $\alpha_3 > \alpha_2$.

With the increase of the applied tension, and thus of the bending angle up to the critical angle $\alpha_{crit} > \alpha_3$, possibly the type ii and iii cracks and the microcracks generated in the type iv tensioned surface will generate type v cracks, not confined cracks that extend along the whole coating, which become evident macroscopically in the surface of the double-dip galvanized coatings. In general, the space between the transverse cracks decreases at this moment, and the type v cracks extend or grow in the longitudinal direction adjacent to

the steel/coating interface, initiating the delamination of the galvanized coating.

As it has been said before, it is estimated an increment of the residual stresses generated in the manufacturing process of double-dip galvanized coatings, with the increase of the total thickness of the coating. For example, it has been observed that for coatings with a total immersion time of 120s, the thickness of the zones is, in general, greater than the thickness of the zones in coatings with total immersion times of 90 s and 60 s; therefore, it is estimated that the possible residual stresses that are generated in each of the zones described in the coatings increase with the thickness of such zones. On the other hand, in the coatings tested by bending, the confined microcracks represent the majority of the cracks observed and described for double-dip galvanized coatings, and these microcracks are the ones that evolve and grow becoming not confined cracks. Even though the density of the confined and not confined microcracks does not vary with the total immersion time in the samples tested up to the critical angle, as has been statistically shown, the thickness of each of the zones of the coatings indeed seem to increase with the immersion time. The thickness of each zone might determine the evolution and growth of the confined cracks, thus determining the critical angle. The microcracks type i, ii and iii might be longer in more tensioned zones for thicker coatings, which would help these to evolve more easily to type v microcracks.

Therefore, it is estimated that, for thicker coatings with possible residual stresses in tension, the confined microcracks evolve and grow until becoming not confined cracks for smaller bending angles, as compared with thinner coatings.

Then, the relative ductility of the double-dip galvanized coatings is significantly influenced by the total thickness of the coatings, and possibly the thickness of the different zones of the coatings and the residual stresses present in each of them have influence on this property, where it is observed a significant decrease of the critical angle as the immersion time increases.

4. Conclusions

The total immersion times utilized in this study are statistically influential in the total thicknesses of the coatings and on the critical angle, but not on the density of the cracks encountered in the samples tested by bending up to the critical angle.

The relative ductility of the double-dip galvanized coatings is influenced by the total thickness of the coatings; possibly the thickness of the different zones of the coatings and the residual stresses present in each of them have influence on this property, where it is observed a significant decrease of 54 % of the

critical angle as the total immersion time increases; the samples coated with a total immersion time of 60 s show the best behavior in the bending test.

References

- [1] R. Parisot, S. Forest, A. Pineau, F. Grillon, X. Demonet, and J.-M. Mataigne, “Deformation and damage mechanisms of zinc coatings on hot-dip galvanized steel sheets: Part i. deformation modes,” *Metallurgical and Materials Transactions A*, vol. 35, no. 3, pp. 797–811, Mar 2004. [Online]. Available: <https://doi.org/10.1007/s11661-004-0007-x>
- [2] E. Tzimas and G. Papadimitriou, “Cracking mechanisms in high temperature hot-dip galvanized coatings,” *Surface and Coatings Technology*, vol. 145, no. 1, pp. 176–185, 2001. [Online]. Available: [https://doi.org/10.1016/S0257-8972\(01\)01323-8](https://doi.org/10.1016/S0257-8972(01)01323-8)
- [3] V. Kuklík and J. Kudláček, “2 - hot-dip galvanizing,” in *Hot-Dip Galvanizing of Steel Structures*. Boston: Butterworth-Heinemann, 2016, pp. 7–16. [Online]. Available: <https://doi.org/10.1016/B978-0-08-100753-2.00002-1>
- [4] A. Marder, “The metallurgy of zinc-coated steel,” *Progress in Materials Science*, vol. 45, no. 3, pp. 191–271, 2000. [Online]. Available: [https://doi.org/10.1016/S0079-6425\(98\)00006-1](https://doi.org/10.1016/S0079-6425(98)00006-1)
- [5] Y. Rico and E. J. Carrasquero, “Microstructural evaluation of double-dip galvanized coatings on carbon steel,” *MRS Advances*, vol. 2, no. 62, pp. 3917–3923, 2017. [Online]. Available: <https://doi.org/10.1557/adv.2017.608>
- [6] S. Ploypech, P. Jearanaisilawong, and Y. Boonyongmaneerat, “Influence of thickness of intermetallic layers on fracture resistance of galvanized coatings,” *Surface and Coatings Technology*, vol. 223, pp. 1–5, 2013. [Online]. Available: <https://doi.org/10.1016/j.surfcoat.2013.02.017>
- [7] ASTM, *ASTM E290 - 14 Standard Test Methods for Bend Testing of Material for Ductility*, ASTM International, West Conshohocken, PA ASTM Std., 2014. [Online]. Available: <http://bit.ly/2ZHgBvY>
- [8] Y. Rico O and E. Carrasquero, “Efecto de la composición química en el comportamiento mecánico de recubrimientos galvanizados por inmersión en caliente: una revisión,” *INGENIUS*, no. 18, pp. 30–39, 2017. [Online]. Available: <https://doi.org/10.17163/ings.n18.2017.04>
- [9] ASTM, *ASTM A123 / A123M-17, Standard Specification for Zinc (Hot-Dip Galvanized) Coatings on Iron and Steel Products*, ASTM International, West Conshohocken, PA Std., 2017. [Online]. Available: <http://bit.ly/2N4adgP>
- [10] C. E. Jordan and A. R. Marder, “Fe-zn phase formation in interstitial-free steels hot-dip galvanized at 450°C: Part i 0.00 wt% al-zn baths,” *Journal of Materials Science*, vol. 32, no. 21, pp. 5593–5602, Nov 1997. [Online]. Available: <https://doi.org/10.1023/A:1018680625668>
- [11] P. Pokorny, J. Kolisko, L. Balik, and P. Novak, “Reaction kinetics of the formation of intermetallic fe - zn during hot - dip galvanizing of steel,” *Metallurgy*, vol. 55, no. 1, pp. 111–114, 2016. [Online]. Available: <http://bit.ly/2XyXZAU>
- [12] S. Ploypech, Y. Boonyongmaneerat, and P. Jearanaisilawong, “Crack initiation and propagation of galvanized coatings hot-dipped at 450°C under bending loads,” *Surface and Coatings Technology*, vol. 206, no. 18, pp. 3758–3763, 2012. [Online]. Available: <https://doi.org/10.1016/j.surfcoat.2012.03.029>
- [13] M. Dutta, A. K. Halder, and S. B. Singh, “Morphology and properties of hot dip zn–mg and zn–mg–al alloy coatings on steel sheet,” *Surface and Coatings Technology*, vol. 205, no. 7, pp. 2578–2584, 2010. [Online]. Available: <https://doi.org/10.1016/j.surfcoat.2010.10.006>
- [14] N. Parvini Ahmadi and E. Rafieezadeh, “Effect of aluminum on microstructure and thickness of galvanized layers on low carbon silicon-free steel,” *International Journal of Iron & Steel Society of Iran*, vol. 6, no. 1, pp. 25–29, 2009. [Online]. Available: <http://bit.ly/2ZBLLVh>
- [15] D. R. Raut and S. H. Poratkar, “Study the effect of aluminum variation on hardness & aluminum loss in zn-al alloy,” *International Journal of Modern Engineering Research (IJMER)*, vol. 3, no. 2, pp. 884–887, 2013. [Online]. Available: <http://bit.ly/2IxBPXc>
- [16] ASM, “Surface engineering” ASM International., 2002. [Online]. Available: <http://bit.ly/2KD2RyT>