

# Tensile behaviour in 30Ni–30Cu–40Mn-based alloys for a dental application

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It was possible to examine the tensile behaviour in experimental 30Ni–30Cu–40Mn-based alloys which were modified by alloying additions of aluminium, indium and tin. Namely, the experimental alloys were developed on the basis of crack formation in a commercial nickel-based alloy and microstructural features in nickel-based alloys investigated. The addition was done by substituting only the manganese content (40 wt%) to 35 and 40 wt%. The results indicated that both changes of tensile strength and elongation were obtained with castability values above 94%. Comparison of tensile properties in experimental Ni–Cu–Mn-based alloys studied here showed that the addition of aluminium to the alloys was appropriate to obtain results similar to those for commercial alloys, indicating that the refining of dendrite arm spacing was obtained by aluminium addition.

## 1. Introduction

The nickel-based casting alloys were crystallized with a well-developed dendrite structure [1, 2], the size of which was affected by the composition of the nickel-based alloys [3–5]. Additionally, the influence of the as-cast structure on the mechanical properties of dental nickel-based cast alloys has been examined to some great extent [5]. It is probable that the morphological features of coarse-grained dendrite structures indicate a smaller tensile strength compared to that of the small-sized ones. Fracture resulting from the applied load normally caused failure either along a grain boundary or within the dendrites [2, 6, 7]. Therefore, it is considered that the microstructure is an important factor in tensile properties during tensile testing. It is also necessary to examine which additive elements are important to the increase in tensile elongation or strength. In this study the formation of a crack due to applied stress was investigated in a commercial nickel-based alloy and the microstructure seen by etching in nickel-based alloys currently on the market, was related to the tensile properties. In additional work, the castability and tensile properties of experimental nickel–copper–manganese-based alloys were evaluated in relation to those of commercial nickel-based alloys.

## 2. Materials and methods

Ni–Cu–Mn-based alloys were made by a vacuum melting the individual elements at  $10^{-5}$  torr ( $1.333 \times 10^{-3}$  Pa): 30 Ni–30 Cu–35 Mn–5 Al, 30 Ni–30 Cu–30 Mn–5 Al–5 Sn, 30 Ni–30 Cu–30 Mn–5 Al–0.1 In–4.9 Sn, 30 Ni–30 Cu–30 Mn–5 Al–2.5 In–2.5 Sn, 30 Ni–30 Cu–30 Mn–5 Al–4.9 In–0.1 Sn and 30 Ni–30 Cu–30 Mn–5 Al–5 In (all wt %). The alloy com-

positions and designations are the same as those used in a previous study on the corrosive properties [8]. They were made using 30 Ni–30 Cu–40 Mn-based alloy with the substitution of Mn, which was called alloy 2 (30 Ni–30 Cu–40 Mn) in our study [7]. The addition of Al, In and Sn elements was done to prevent the formation of copper oxide during melting and casting. On the other hand, six commercial Ni-based alloys were used, indicating that they had typical properties for crown and bridgework [9–12].

The properties investigated in this study were examined by the following schedules.

1. The formation of cracks was clarified using the cast specimen (a commercial Ni-based alloy; Fittlog 50 TYPE 1, Fittlog investment) indicated in Fig. 1. In tensile-testing of the alloys which were cast by air-pressure (Fitter) after torch-melting, crack formation was observed, and the castability values were evaluated using the specimens cast by air-pressure and centrifugal castings (Castron-8).

2. On the basis of their results, typical Ni-based alloys were examined for microstructural features, Vickers hardness and tensile properties.

3. The experimental Ni–Cu–Mn-based alloys were compared for castability, microstructural features and tensile strength with commercial Ni-based alloys tested as above.

The mechanical properties, such as Vickers hardness, tensile strength and castability, were also measured. The hardness values were those when a 200 g load was applied to the specimen (15 mm × 20 mm × 2.5 mm). The tensile specimens had a diameter 1.5 mm and gauge length 15 mm, and three duplicate specimens were obtained within the investment for every casting. The tensile conditions used were as reported previously [6, 7]. The castability

values were evaluated for two coil-like specimens with diameter 1.0 mm for spiral (Type I) and 0.7 mm for both sprue canal and spiral (Type II) with a ratio of  $M_{\text{ingot}}/M_{\text{coil}}$ , where  $M_{\text{ingot}}$  is the weight, 3 or 6 g, used for air-pressure or centrifugal castings, and  $M_{\text{coil}}$  is the weight of the specimen which was cast perfectly under four rotating times for a wax spiral with different diameters of 1.0 and 0.7 mm. The reported values for each measurement were means and standard deviations of 10–12 indentations. During casting the investments used were gypsum-bonded Fittment investment (Code FM), two kinds of phosphate-bonded investments such as Crownvest investment (Code CV) and Univest Nonprecious investment, also used for the other Ni-based alloys including ternary Ni–Cu–Mn-based alloys).

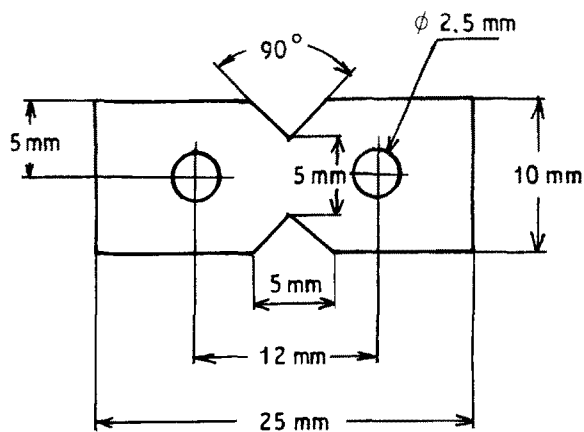


Figure 1 Tensile specimen for observation of crack formation.

TABLE I Tensile properties of a commercial nickel-based alloy (Fittloy 50 TYPE 1) when the casting conditions were different (FV, CV and SV = investments used; see text)

Casting machine and investments		Fracture strength ( $\text{kg mm}^{-2}$ )	Elongation (%)
Pressure casting	FM	$21.7 \pm 1.8$	$0.8 \pm 0.1$
Centrifugal casting	CV	$32.4 \pm 2.9$	$1.3 \pm 0.2$
	SV	$22.7 \pm 1.2$	$1.2 \pm 0.1$

### 3. Results

Using a tensile specimen with two V-shaped notches (Fig. 1), the tensile properties indicated in Table I were measured. The as-cast microstructures obtained by air-pressure casting were dendrite structures, as shown in Fig. 2a (FV investment) and b (CV investment). After tensile testing with 0.015 and 0.020 tensile strain, the formation of cracks marked by a “V”, in tensile specimens which were cast by air-pressure, were observed (Fig. 3a and b). The castability value in centrifugal casting was remarkably larger than that in air-pressure casting (Fig. 4). From this result the low-melting nickel-based alloys were centrifugally cast in this study. The tensile specimens in a commercial nickel-based alloy had the values of  $21.7\text{--}32.4 \text{ kg mm}^{-2}$  (tensile strength) and about 1% (elongation). According to the testing schedule, the cast microstructures in commercial Ni-based alloys were observed to be comparable with experimental Ni–Cu–Mn-based alloys (Figs 5 and 6). Their alloys showed Vickers’ hardness values of 154–274 (Table II). In this study the alloys with a hardness above 220 were considered to be hard [13–15]. Fig. 7 shows the tensile behaviour in alloys S1, S2 and S3 deformed to tensile elongations of 2.5%–22.1%, whereas alloys H1, H2, H3 had tensile strength below  $35.0 \text{ kg mm}^{-2}$  and tensile elongation below 1.1%, as shown in Fig. 8 (Table III).

The Ni–Cu–Mn-based alloys cast by centrifugal casting were castable similar to a control material with  $90.4 \pm 4.4\%$  (Type I) and  $66.4 \pm 1.5\%$  (Type II). In particular, the Type II coil-like specimen with a 0.7 mm diameter spiral also showed better castability (Table IV). The tensile properties changed remarkably with the addition of alloying elements to the ternary Ni–Cu–Mn alloy (Table V). In stress–strain curves (Fig. 9), the Ni–Cu–Mn-based alloys fractured at some elongation after a linear increase of tensile strength. The value of tensile strength at fracture (fracture strength) was larger in the 30 Ni–30 Cu–35 Mn–5 Al alloy than the others, together with a larger elongation to fracture. The dendrite arm spacing in these alloys ranged from 4–12  $\mu\text{m}$  average value (Table VI).

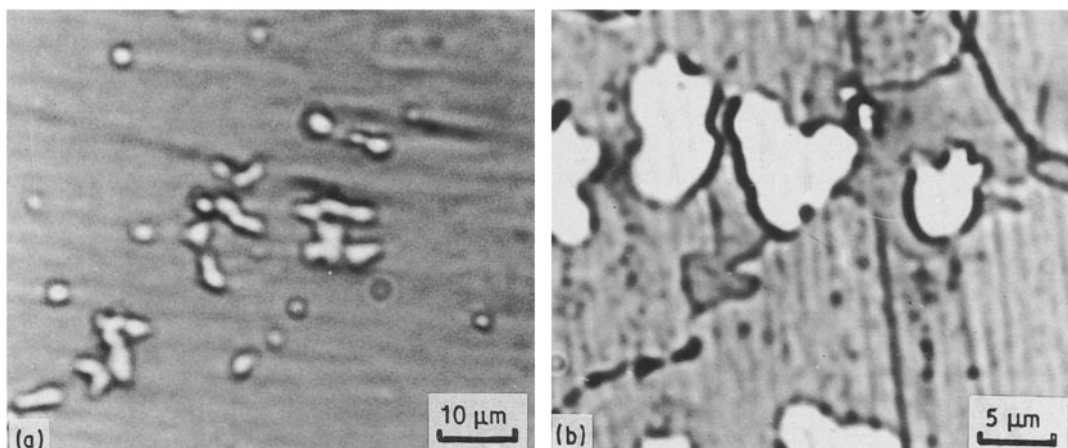


Figure 2 Microstructures of a commercial nickel-based alloy (Fittloy 50, TYPE 1) cast by (a) air-pressure (Fittment investment) and (b) centrifugal castings (Crownvest investment).

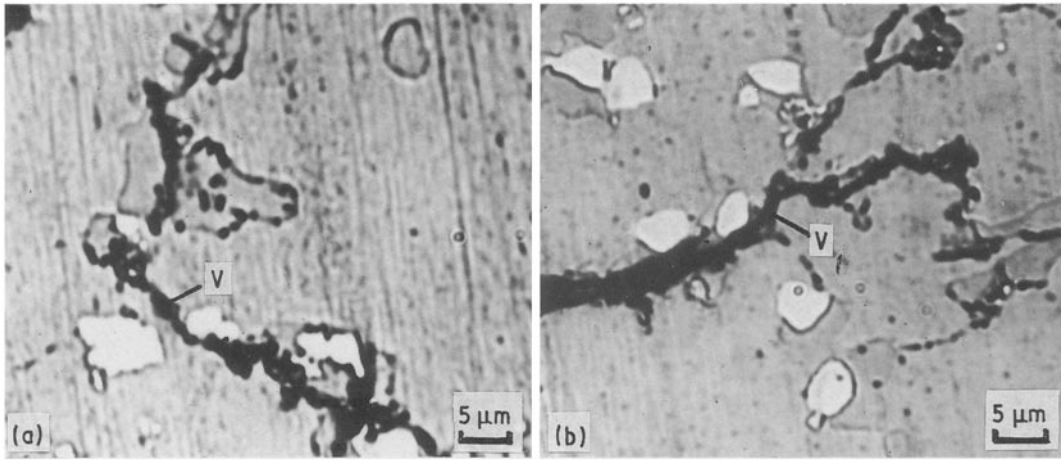


Figure 3 Microstructures including crack formation sites (V, dark area) when the cast specimens (air-pressure casting) were tested by (a) 0.015 and (b) 0.020 tensile strain.

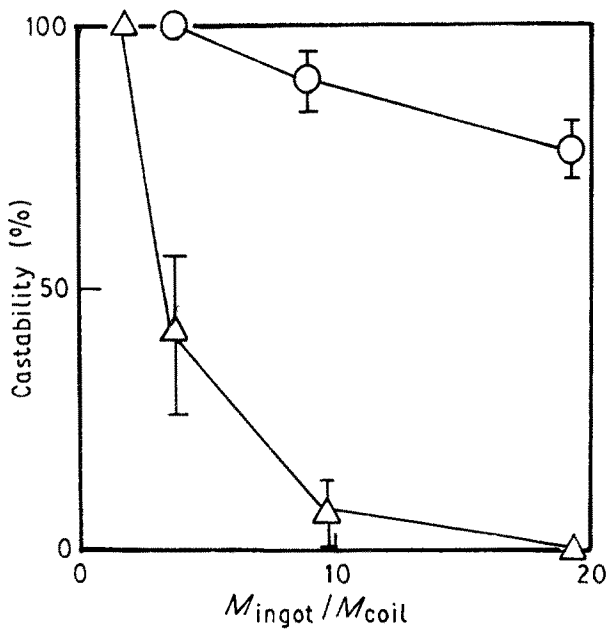


Figure 4 Variation of castability values with the ratio  $M_{ingot} / M_{coil}$  in the commercial low-fusing alloy ( $\Delta$ ) air-pressure, ( $\circ$ ) centrifugal casting.

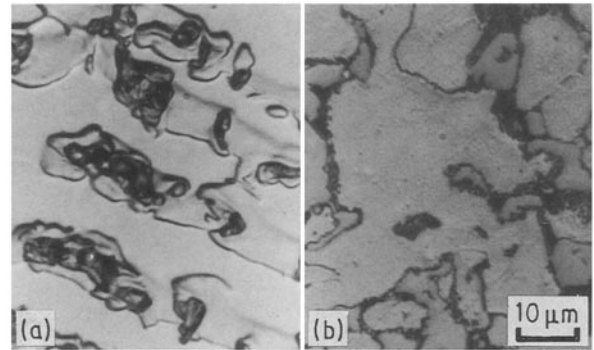


Figure 6 Microstructures in as-cast specimens: (a) H1, (b) H3 (for key, see Table II).

#### 4. Discussion

The Ni–Cu–Mn-based alloys had the same castability as the ternary Ni–Cu–Mn alloys [9], and the values of castability in the Type I coil-like specimen were above about 94% (Table IV). Their values were almost the same as a control material (Fittloy 50 Type 1), while the Type II coil-like specimen with a spiral of diameter

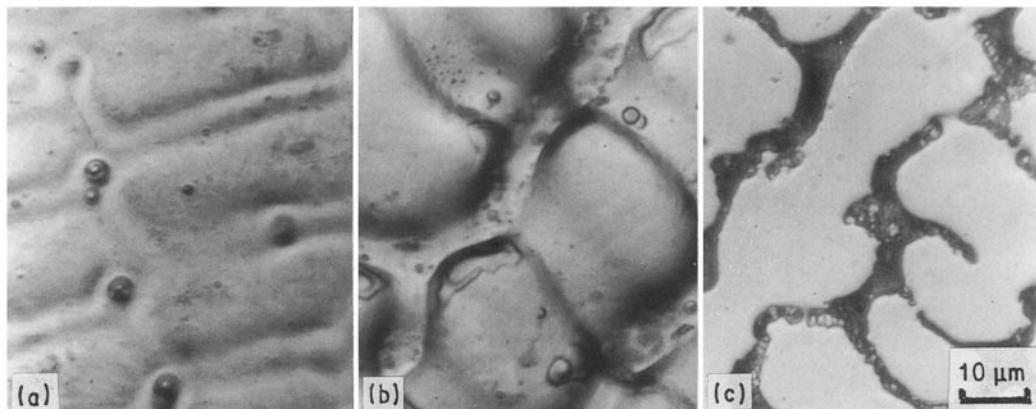


Figure 5 Microstructures in as-cast specimens: (a) S1, (b) S2, (c) S3 (for key, see Table II).

TABLE II Hardness values in six commercial nickel-based alloys investigated (see text)

Code	Hardness (H <sub>v</sub> )	Brand name	Manufacturer
S1	154 ± 2	Summalloy Nickel soft	Shofu Inc
S2	168 ± 4	Crown-8	Sankin Ind.
S3	200 ± 5	Vatacoballium soft	Yata Chem. Co.
H1	231 ± 5	Taicrown	GC Co.
H2	249 ± 9	Fittloy 50 TYPE 1	Sankin Ind.
H3	274 ± 5	Adcast	Nihon Siken Co.

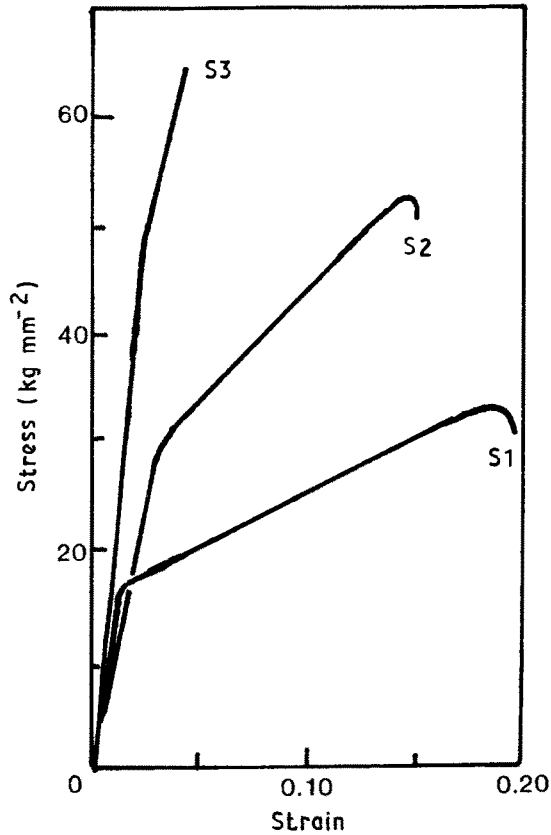


Figure 7 Stress-strain curves in commercial nickel-based alloys S1, S2 and S3 when they were deformed to fracture in the tensile test.

TABLE III Fracture strength and elongation of the commercial alloys in tensile testing

Code	Fracture strength (kg mm <sup>-2</sup> )	Elongation (%)
S1	35.0 ± 2.0	22.1 ± 4.0
S2	42.2 ± 2.2	12.3 ± 0.7
S3	55.5 ± 4.2	2.5 ± 0.2
H1	34.5 ± 2.1	1.1 ± 0.1
H2	21.7 ± 1.8	0.8 ± 0.1
H3	34.3 ± 2.1	0.4 ± 0.0

0.7 mm showed values above 50%. Thus their alloys could be applied to dental purposes such as crowns and bridges because of their better castability. The results of tensile properties could also support these applications, because experimental alloys with greater deformability than some alloys having less deformation, were obtained (Tables II and V). The observed

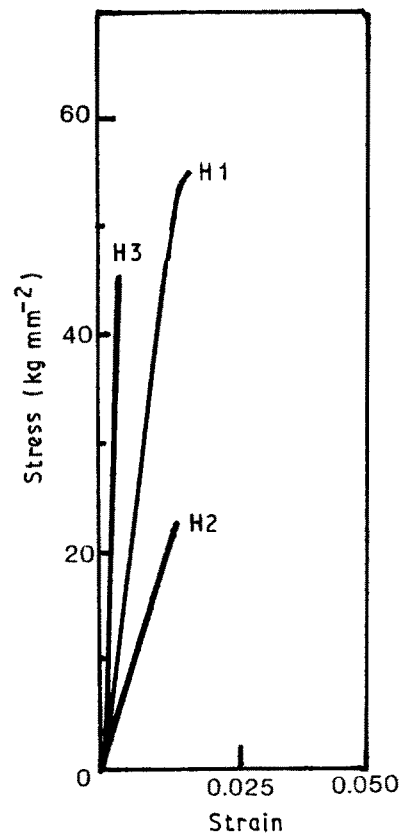


Figure 8 Stress-strain curves to tensile fracture of commercial nickel-based alloys H1, H2 and H3.

TABLE IV Castability values in type I and type II coil specimens in experimental alloys tested

Materials	Castability (%)	
	Type I	Type II
30 Ni-30 Cu-35 Mn-5 Al	100	55.3 ± 8.9
30 Ni-30 Cu-30 Mn-5 Al-5 Sn	94.3 ± 4.9	31.3 ± 7.9
30 Ni-30 Cu-30 Mn-5 Al-0.1 In-4.9 Sn	94.0 ± 8.5	56.7 ± 1.0
30 Ni-30 Cu-30 Mn-5 Al-2.5 In-2.5 Sn	93.7 ± 2.1	58.0 ± 9.5
30 Ni-30 Cu-30 Mn-5 Al-4.9 In-0.1 Sn	100	65.0 ± 0.5
30 Ni-30 Cu-30 Mn-5 Al-5 In	98.7 ± 2.3	35.0 ± 5.1

TABLE V Tensile properties in experimental alloys tested

Materials	Fracture strength (kg mm <sup>-2</sup> )	Elongation (%)
30 Ni-30 Cu-35 Mn-5 Al	36.2 ± 5.1	3.0 ± 0.3
30 Ni-30 Cu-30 Mn-5 Al-5 Sn	8.5 ± 2.1	0.7 ± 0.1
30 Ni-30 Cu-30 Mn-5 Al-0.1 In-4.9 Sn	10.6 ± 1.9	1.1 ± 0.4
30 Ni-30 Cu-30 Mn-5 Al-2.5 In-2.5 Sn	13.5 ± 2.3	2.0 ± 0.5
30 Ni-30 Cu-30 Mn-5 Al-4.9 In-0.1 Sn	9.1 ± 3.6	1.5 ± 0.5
30 Ni-30 Cu-30 Mn-5 Al-5 In	8.3 ± 1.3	0.9 ± 0.1

tensile properties were related to microstructural variations which were produced as a result of the manipulative changes due to casting conditions [6, 7, 16, 17]. Thus, the conditions chosen were the same as those reported previously [6, 7]. In this study, the difference between casting machines (air-pressure casting and centrifugal casting) was significant for FM

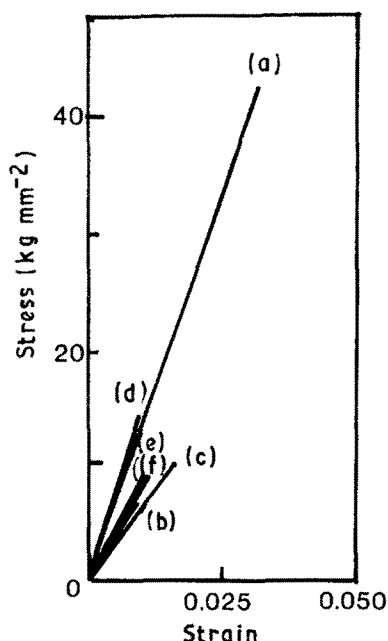


Figure 9 Stress-strain curves in tensile testing: (a) 30 Ni-30 Cu-35 Mn-5 Al, (b) 30 Ni-30 Cu-30 Mn-5 Al-5 Sn, (c) 30 Ni-30 Cu-30 Mn-5 Al-0.1 In-4.9 Sn, (d) 30 Ni-30 Cu-30 Mn-5 Al-2.5 In-2.5 Sn, (e) 30 Ni-30 Cu-30 Mn-5 Al-4.9 In-0.1 Sn, (f) 30 Ni-30 Cu-30 Mn-5 Al-5 In.

TABLE VI The values of dendrite arm spacing in 30 Ni-30 Cu-40 Mn-based alloys investigated

Materials	Dendrite arm spacing ( $\mu\text{m}$ )
30 Ni-30 Cu-35 Mn-5 Al	$4 \pm 1$
30 Ni-30 Cu-30 Mn-5 Al-5 Sn	$5 \pm 1$
30 Ni-30 Cu-30 Mn-5 Al-0.1 In-4.9 Sn	$7 \pm 1$
30 Ni-30 Cu-30 Mn-5 Al-2.5 In-2.5 Sn	$12 \pm 2$
30 Ni-30 Cu-30 Mn-5 Al-4.9 In-0.1 Sn	$10 \pm 1$
30 Ni-30 Cu-30 Mn-5 Al-5 In	$10 \pm 2$

and CV investments ( $p < 0.01$ ), but no significance between FM and SV investments was found. The phosphate-bonded investments (CV and SV) had different size distributions of silica particles, indicating that the former had a smaller size (below  $37 \mu\text{m}$ ; 36%) and a larger size ( $150\text{--}420 \mu\text{m}$ ; 39%) and the latter had an intermediate size ( $53\text{--}149 \mu\text{m}$ ; 65%) [18]. These characteristics would affect the escape of gases from the investment mould. The main factor in the investment affecting the tensile properties of the alloys is considered to be a casting defect such as porosity, [16] and a porous specimen was fractured immediately after tensile testing [19]. The elongation (0.9%–32.6%) and tensile strength ( $32\text{--}66 \text{ kg mm}^{-2}$ ) in 80 wt % Ni-20 wt % Cr alloy were evaluated, indicating that the porosity was below 2.9% (average value). In Fig. 2, no difference in the microstructures containing a grain of dendritic shape (white areas on the optical micrographs) can be seen and no fracture after initiation of tensile test was found. It seems that the additive element plays the role of a deoxidant without forming a large porosity in tensile specimens. As expected, the higher mechanical strength of the alloys tested would

be associated with the absence of porosity, especially when improvement in the alloys could be achieved by adding appropriate elements or a combination of them.

During the initial solidification of nickel-based alloys, dendrite structures were formed (Figs 5 and 6). The microstructural features are changed by the individual alloying elements within the alloy formulation. Dendritic crystallization was dependent upon solidification rate and alloy compositions [20–22]. In the Ni-Cu-Mn-based alloys investigated, the hardness value was increased by additional alloying additives to the ternary Ni-Cu-Mn alloy, because the matrix phase was hardened by both additives, such as a single element (either Al or In), and an additive compound (Al and In; Al-In, and Al, In and Sn; Al-In-Sn) [23]. This hardened matrix did not correspond to an increase in fracture strength. When additives such as In and Sn were used for 30 Ni-30 Cu-40 Mn-based alloys, the matrix strength decreased, and the elongation of the matrix ranged approximately from 0.7%–2.0% (Table V). In Ni-Cr-based alloys reported previously [1–5, 16], their alloys contained molybdenum, silicon and carbon as additive elements, and the melting temperature was found to be increased in comparison with binary Ni-Cr-based alloys without additives. Thus, additives with low-melting temperatures below  $1000^\circ\text{C}$  must be selected, as indicated in this study. The addition of Al as a single additive was effective for the Ni-Cu-Mn-based alloy, giving increases in both fracture strength and elongation, compared with the other alloys used in this study. The ternary 30 Ni-30 Cu-40 Mn alloy had a fracture strength of  $30.38 \pm 4.83 \text{ kg mm}^{-2}$  with an elongation of  $9.87\% \pm 7.32\%$  [7], and the single additive Al changed the microstructure to dendritic from cellular [8, 24]. The average dendritic arm spacing was then  $4.0 \mu\text{m}$ , the smallest value of all the alloys tested, as indicated in Table VI, the value indicating the refining of dendrite structure.

Further supplementary results are needed to define alloy composition for strengthening of the matrix phase. This study details only the tensile behaviour in experimental 30 Ni-30 Cu-40 Mn-based alloys with different dendrite arm spacings, and indicates that a single element (Al) of the additives investigated would be effective in improving the alloys because of the small-scale dendrite spacing obtained. Stress analysis revealed that the alloying additive could be selected adequately to change the ternary alloy to a better alloy.

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