Topological dependence of mechanical responses of solidification microstructures in aluminum brazed joints [①]

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Abstract: The main objective is to provide an evidence of spatial dependence of mechanical responses of a heterogeneous aluminum brazed joint re-solidified clad, and to confirm a sufficient sensitivity of a nano-indentation —load curve method for identifying the dependence. Topological features of a network of solidification microstructures(α phase and eutectic), formed during quench in a brazing process of aluminum alloy, influence significantly dynamic mechanical responses of re-solidified heterogeneous material. Nano/ micro indentation depth vs load characteristics of differing phases suggest a spatially sensitive mechanical response of a re-solidified fillet in the joint zone. Hence, a spatial distribution, pattern formations and other morphological characteristics of microstructures have a direct impact on an ultimate joint integrity. Topology-induced variations of indentation —load curves was presented. A hypothesis involving microstructures' spatial distribution vs mechanical response was formulated.

Key words: nano-indentation; solidification microstructure; aluminum brazing; mechanical response

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1 INTRODUCTION

Mechanical integrity of an aluminum brazed joint formed from a brazing sheet or a filler metal exposed to controlled atmosphere process conditions significantly differs from the features that may be assessed analyzing bulk properties of cladding/ filler metals. This is because: 1) a significant alloying within joints taking place; 2) new phases formed promoting embrittlement; 3) the morphology of microstructures dramatically affected by quench conditions and 4) stress conditions differing from the preceding ones[1]. The integrity of a joint is usually analyzed a posteriori (i e, not predicted) and an empirical input is a must in assessing the influence of process parameters vs joint characteristics. One additional problem becomes increasingly more important. The significance of influence of a solidification microstructures' pattern and topological characteristics of the resulting structure on ultimate properties of a heterogeneous material promotes a need for an exploration of the means to control the properties at the nano/ micro level. The fundamental problem one may encounter is related to the fact that solidification microstructures, say an α phase dendrite imbedded in irregular eutectic, represent very small-volume objects, not easily accessible to traditional measurements of mechanical characteristics.

One of the techniques developed(in particular for thin film structures), is the nano-indentation vs load curve dynamic evaluation, which can provide a valuable information about properties of thin film systems and/ or small volume sub-structures[2−7]. This method was used in this research to gain an insight into nonuniformity of mechanical responses of solidification microstructures formed within the re-solidified Al+ Si sub-eutectic fillet under conditions of controlled atmosphere brazing of AA4343/ AA3003 brazing sheets.

To the best knowledge of the authors, brazed joint's nano-mechanical responses involving sub-structures thereof have not been studied by using dynamic nano-indentation techniques. The re-solidified material in the joint zone is heterogeneous, so the depth-sensing indentation tests are applied similar to the technique used recently for analysis of the rate dependent indentation characteristics of some solder alloys[8]. More details about the test procedure used can be found in Ref. [9].

2 EXPERIMENTAL

Two kinds of cladding sheet materials identified as material A and B in this paper were used to prepare the samples. Chemical compositions of these two ma-

terials are listed in Table 1. The dimensions of the coupon were 55 mm × 25 mm × 0. 35 mm(plate). In addition, the wedge T samples were assembled, with a vertical piece(made of AA3003 alloy) located along the mid plane of the above-mentioned plate and with its dimensions as follows: 55 mm × 25 mm × 1. 0 mm. In all the coupons, the cladding ratio of these brazing sheets was around 10%. The substrate material of the cladding sheets was also AA3003.

Samples were exposed to a series of heating and/ or brazing cycles in atmosphere of ultra high purity N_2(99. 999%) in a tightly controlled CAB (Controlled Atmosphere Brazing) furnace. The peak brazing temperature was attained with a very good stability of ±0. 5 K at 800 K. The uncertainty in determining temperatures was less than ±0. 5 K with a resolution of 0. 1 K. The brazing cycles were conducted at 873 – 893 K, as illustrated in Figs. 1 and 2[9]. The ramp-up heating rate and rapid quench were the same in all experiments(with 80 K/ min during the ramp up and 90 K/ min during quench respectively). Brazing experiments were performed using potassium fluoraluminate as flux.

The samples were cut and mounted at room temperature in order to avoid significant deformation. Polishing was performed using the standard procedures. After etching by Keller's solution(2 mL HF + 3 mL HCl + 5 mLHNO$_3$ + 190 mL H$_2$O), the samples were observed using Nikon Epiphot

Table 1　Chemical composition of materials (substrate AA3003/ clad A or B AA4343, mass fraction, %)

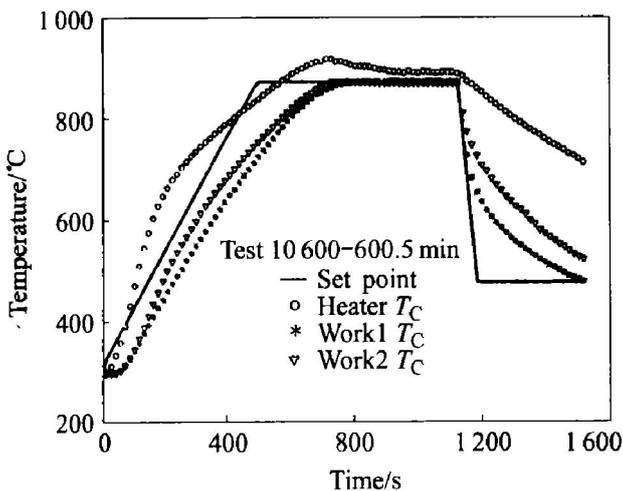| Material	Si	Mn	Fe	Cu	Mg	Cr	Al
Substrate	0. 6	1. 2	–	< 0. 2	–	–	Bal.
Clad A	6. 523	0. 002	0. 133	0. 003	0. 005	0. 001	Bal.
Clad B	7. 192	0. 015	0. 160	0. 27	0. 004	0. 001	Bal.

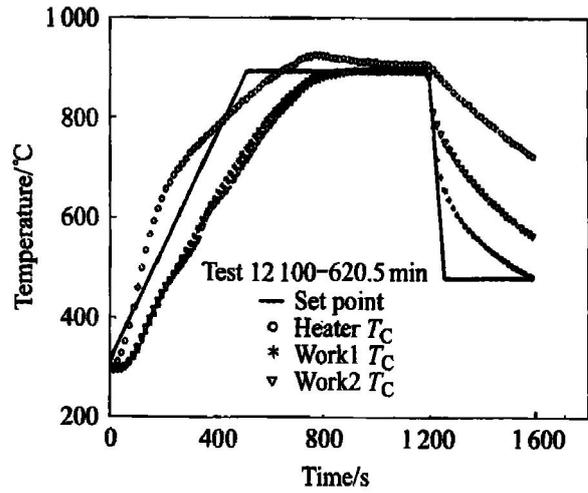**Fig. 1**　Curves of temperature vs time (T_{peak}= 873 K, dwell time 5 min)

Fig. 2　Curves of temperature vs time (T_{peak}= 893 K, dwell time 5 min)

300 inverted optical metallurgical microscope. The image processing was conducted using Image-Pro Plus software. The nano-indentation tests were performed at room temperature using a Micro Zone Tester (Akashi Co, Japan) equipped with a Berkovich indenter.

3　RESULTS AND DISCUSSION

3. 1　Mechanical property response of heterogeneous joint zone

In Fig. 3, an illustration of a mechanical response of a heterogeneous material of the joint zone is presented. Nano-indentation curves are obtained for a series of different brazed joints. Two of them (with a designator 873, Fig. 3) were obtained by quenching a corresponding wedge T sample made of AA3003/ AA4343(horizontal) and AA3003(vertical) mating surfaces from 873 K peak brazing temperature. The remaining two others(with a designator 893) were quenched from 893 K. Designator A and B represent the clad material in Table 1, and L denotes one(left) side of brazed joint.

From Fig. 3, it should be noticed that the samples quenched from a lower temperature contains less fraction of α phase dendrites than the ones quenched from a higher temperature does. In order to reveal the bulk mechanical response, the relatively larger load, 500 mN was utilized to illuminate the influence of topology of brazed joint. That is, the indentation area in brazed joint will be the mixture of alpha and eutectic phases. From the corresponding mechanical response, it is clear that the former indicates a harder heterogeneous material as a whole. In accordance with that conclusion, it must be clear that it features a harder matrix with a sparsely distributed softer alpha phase dendrites. This conclusion would be confirmed if one would compare the two differently manufactured materials quenched from the same tempera-

ture(i e, the pairs of samples with designators A-873 vs B-873, as shown in Fig. 3). The heterogeneous structure with a very little distributed alpha phase(B-873) is harder when compared with the one with more pronounced presence of alpha phase (A-873). Why these materials feature different population of the alpha phase is an issue to be discussed elsewhere[10].

3. 2　Spatial dependence of mechanical responses of individual phases

In Fig. 4, typical nano-indentation impressions are illustrated. The nano-indentation test results performed on a sample obtained after quenching from 873 K is presented in Fig. 5. A striking feature of mechanical responses is that a location of the indentation dramatically influences the mechanical response of the structure. There is a pattern that can easily be identified. Locations near the clad-core interface feature a response that resembles a harder eutectic. In contrast, locations closer to the equilibrium free surface membrane feature a softer response. The presence of alpha phase in the surroundings of a test location leads to a less hard response. Such a clear topological dependence of mechanical responses is present, despite of an application of very small loads and nano scale indentations. It is very indicative to notice that even if the boundary conditions' (i e, size effect's) influence is assumed to be similar in various cases(the responses for positions 2 and 4, in Fig. 5), the response of the same structure would still feature sizable difference in the mechanical response when compared to the ones noticed elsewhere. Therefore, we may hypothesize that in addition to boundary conditions (which obviously have an influence), a spatial distribution of the phases in a re-solidified clad during joint formation has an influence on the mechanical response.
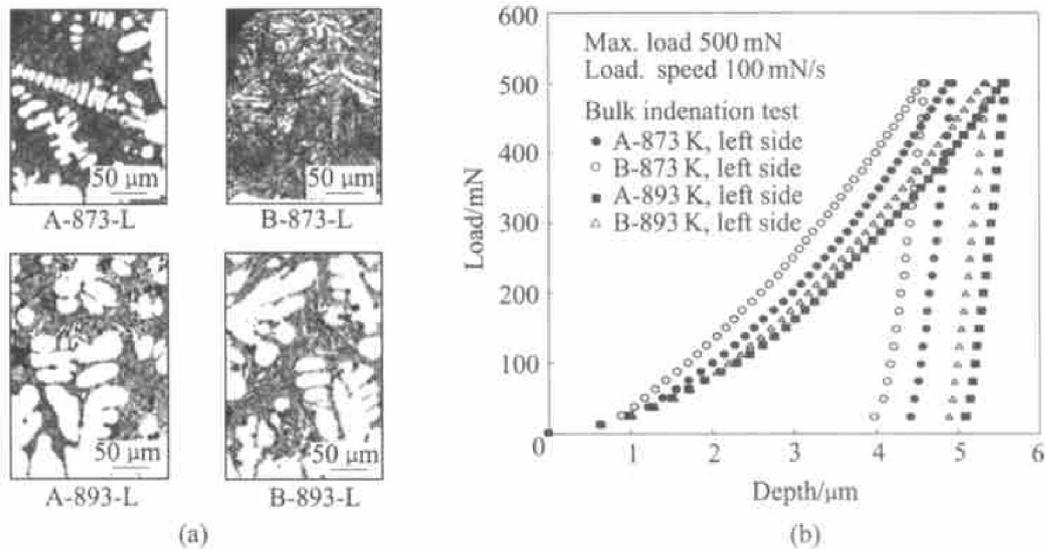
To further analyze the above formulated hy-

Fig. 3　Indentation —load curves for heterogeneous material and bulk mechanical response of brazed joint zone
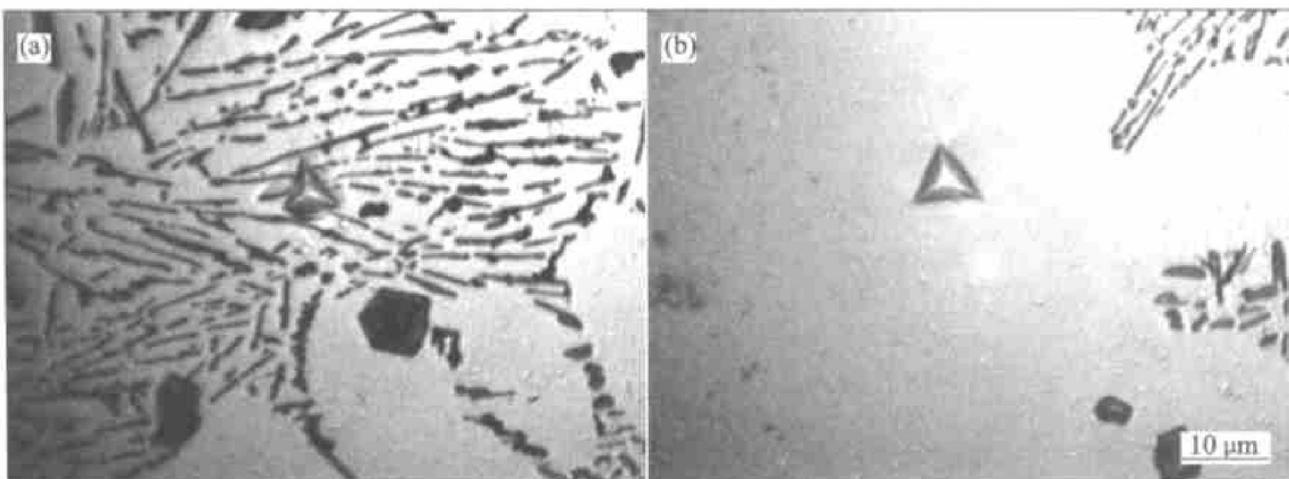(a) —Indented materials; (b) —Indentation test results

Fig. 4　Typical impressions of indentation
(a) —Eutectic phase; (b) —Alpha phase

pothesis, let us consider an influence of microstructure location within the heterogeneous matrix on the mechanical response of the alpha phase. In Fig. 6, several locations within a joint zone formed after quenching from 893 K are selected for comparison. Table 2 provides data on experimental conditions corresponding to indentation—load curves presented in Fig. 6. The alpha phase in microstructures are marked on a micrograph in the inset. It is interesting to notice the following phenomena: 1) the nano-response is location sensitive; 2) if the alpha phase microstructure is fully surrounded by hard eutectic matrix, the response is not significantly location-dependent; 3) if the alpha phase is not fully surrounded by eutectic matrix, the mechanical response differs more significantly. These observations are preliminary and a

further detailed study of these observations is under way. In any case, it is obvious that the nano-indentation response does feature some spatial characteristics influenced by joint topology. Such behavior clearly influences the micromechanical response of the heterogeneous material as presented in Fig. 3.

Table 2 Typical indentation parameters (adopted in Fig. 6)

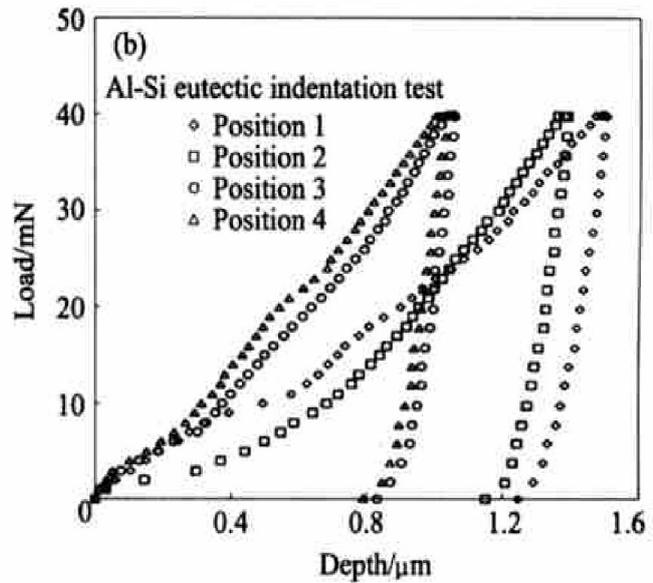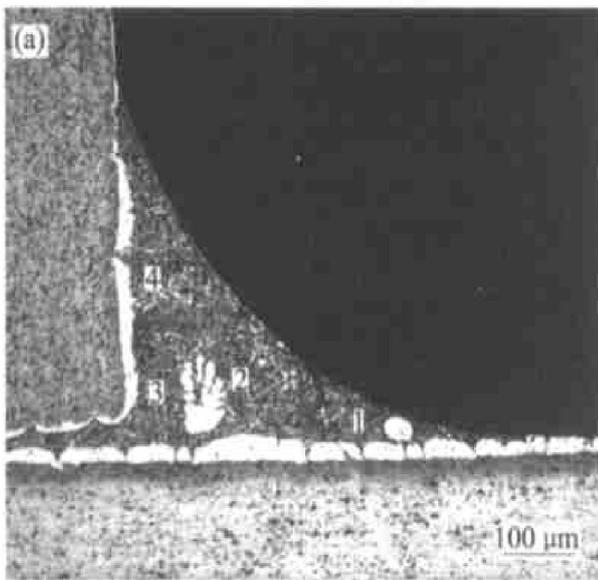| Position	Loading speed/ ($mN \cdot s^{-1}$)	Max. Load/ mN	Hold time/ s	Unload time/ s
A				
B	5	20	1	1
C				
D				

Fig. 5 Curves of load vs nano-indentation for irregular eutectic joint area
(sample B-873-R quenched from 873 K)
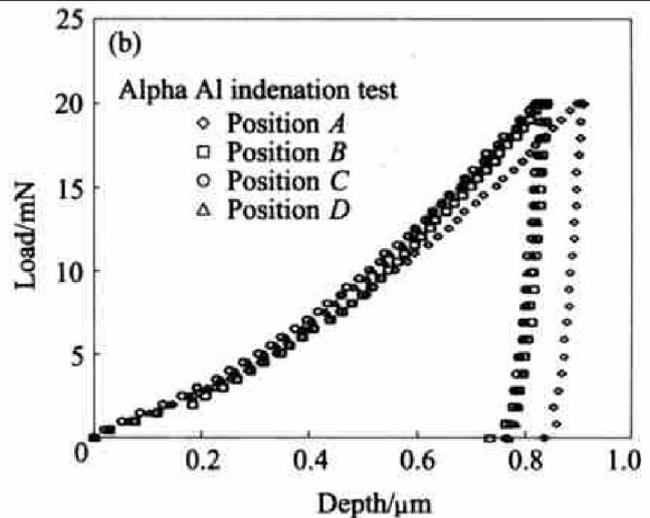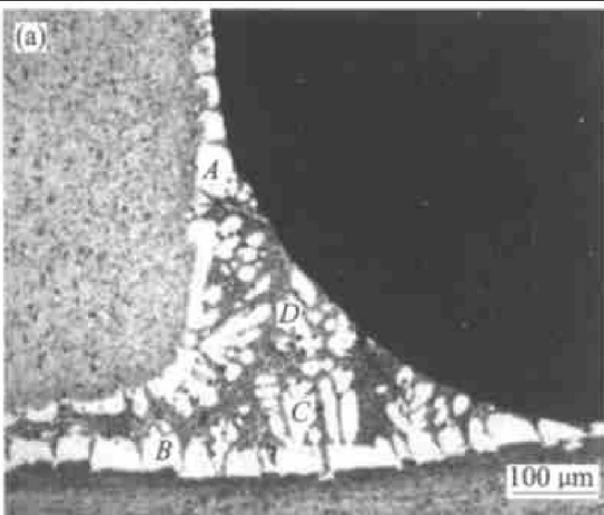(a) —Indented material; (b) —Indentation test results

Fig. 6 Curves of load vs nano-indentation for alpha phase
(sample A-893-R quenched from 893 K)
(a) —Indented material; (b) —Indentation test results

4　CONCLUSIONS

1) The nano indentation — load curve dynamic response method does provide a sensitive tool for investigating the mechanical response of solidification microstructures formed in an Al + Si system during brazing.

2) Nano mechanical responses of solidification microstructures are spatially sensitive. That is, the microstructures(alpha phase or eutectic phase) show different mechanical responses due to different locations in the brazed joint domain, resulting in a sizable difference of the mechanical properties of the heterogeneous material within the brazed joint.

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