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Effects of Neutron Irradiation on Non-fissionable Alloys*

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ABSTRACT

Neutron irradiation can cause three major effects in non-fissionable alloys: 1) the excess vacancies produced by irradiation can accelerate diffusion; 2) point defects and regions of strain can serve as additional nucleation sites for phase changes; 3) the displacement of atoms may cause a breakup of ordered or precipitated regions. By the proper choice of alloy systems, the first two effects have been isolated and examined in detail.

Essentially pure enhanced diffusion can be achieved in short-range ordering alloys such as the α -phase of Cu-Zn and Cu-Al. A quantitative theory was developed which shows that the temperature and flux dependence of enhanced diffusion varies with the relative amounts of excess vacancies and of their trapping sites in the solid. The predictions of the theory for high trapping site concentration have been verified in Cu-Zn by neutron irradiation, and those for the low trapping site concentration by positive ion bombardment. Experiments on Cu-Al indicate that the low trapping site case can be achieved in this alloy with neutrons. These experiments also show that by controlled irradiation an alloy can be put into a low temperature state which cannot be achieved by thermal means alone.

The change in the number of nucleation sites has been studied in the iron-carbon system. Since the carbon diffuses interstitially the diffusion rate cannot be accelerated, and no precipitated regions exist in freshly quenched specimens. Therefore, all changes in the rate of disappearance of

carbon from solution must be related to trapping, viz., nucleation phenomena. After a few hours irradiation at ambient temperature, internal friction measurements of the carbon peak showed that it disappears ten times faster than the thermal rate. The implication that ten times the nuclei are present is confirmed by electron microscope studies. After several days irradiation at low temperature, however, the rate of disappearance of carbon from solution is one thousand times the thermal rate and is governed precisely by the activation energy for diffusion of carbon in iron. Detailed studies of this phenomenon with electrical resistivity measurements show several decay steps which are related to the interaction of the carbon atoms with point defects.

A. Effects of Radiation of Alloys

The study of radiation damage in solids was originally motivated by a concern for the property changes to be expected in reactor materials. However, in recent years this type of study has been of increasing importance to the investigation of solid state phenomena in non-reactor materials, since fast particle bombardment is an excellent method for the introduction of controlled numbers of defects into a solid. The control over the type and distribution of the defects is particularly important in alloys, because any given thermodynamic state of an alloy is characterized by a positional relationship of the atoms. This relationship can be altered in three important ways by heavy particle bombardment.

1. Destruction of ordered or precipitated regions. Theoretical studies of the details of damage in a solid by a highly energetic primary knock-on indicate that near the end of its path the knock-on displaces a large number of atoms (1), and also causes interchange of atoms by replacement collisions. Although many of these displaced atoms may relax back to their original positions, the periodicity of any ordered structure which may have existed in this region will have been partially destroyed. If severe local damage occurs near a precipitate some of the clustered atoms should be driven back into solution. The destruction of order by neutron irradiation has been studied in Cu_3Au (2) and Ni_3Mn (3) alloys, and the breakup of precipitates has been reported for both proton (4) and neutron (5) bombardment of Fe-Cu.

2. Enhanced nucleation. The local damage region at the end of the path of the primary knock-on might be expected to serve as a nucleation

site for phase changes. Either the large strains arising from the displaced atoms could be relieved by the formation of a new phase in the vicinity, or the voids which must also be present in this same region of large damage could release the lattice strains surrounding a precipitating atom. A study of the white to gray tin transformation has demonstrated the enhancement of nucleation (6). White tin is unstable below 12°C but the transformation to gray tin is exceedingly slow unless the white tin is either seeded or severely cold worked. Prior low temperature irradiation of the white tin in a reactor will also induce the transformation, which indicates that either the strains of the damaged regions are equivalent to cold work strains or that some of the atoms in the damaged regions have rearranged themselves into the gray tin structure. Regardless of the details of the mechanism, neutron radiation has enhanced the nucleation of the phase change.

3. Enhanced diffusion. In a large class of metals and alloys atomic diffusion takes place by the vacancy mechanism. The addition by irradiation of vacancies in excess of the number present in thermal equilibrium will, therefore, accelerate diffusion. It has been shown that the diffusion coefficient associated with irradiation in the average reactor is only of the order of 10^{-20} cm²/sec (7). Since such small diffusion coefficients of tracer atoms cannot as yet be observed, enhanced diffusion is not yet accessible to measurement in pure metals. However, the migration of vacancies in an alloy can cause a large number of local atomic interchanges which can be detected in the changes of some physical properties such as electrical resistivity. This type of measurement has been made on several alloy systems, some of which will be reviewed by

M. Wechsler in this Symposium.

If the bombarding particle either has a low energy or is light in mass (electrons or γ -rays), the severe local damage and replacement collisions discussed above are not expected. Only the simple displacement of atoms occurs and, therefore, only single vacancy-interstitial pairs are created. As far as is known no destruction of order or creation of nucleation centers occurs with electron or gamma irradiation and, therefore, only the enhanced diffusion effect is expected. Since during the irradiation of many alloys at least two of the above effects are occurring simultaneously, it is important to find ways of isolating the individual effects for a quantitative study of each. Two of these effects, enhanced diffusion and enhanced nucleation, have been isolated and studied separately. Investigation of these two effects are discussed in this paper.

B. Enhanced Diffusion

Enhanced diffusion can be studied by either using only electron or γ -ray radiation or by choosing an alloy system in which enhanced diffusion is the dominating factor. We wanted to make a quantitative study of enhanced diffusion under all types of radiation and, therefore, chose an alloy, Cu-brass, in which this is the major effect. This alloy has proved to be a fortunate choice for these studies in many respects (8). It does not become unmanageably radioactive after neutron irradiation; the slight preference of the Cu-Zn bond causes short-range ordering without the development of long-range order; equilibrium short-range order is achieved in reasonable lengths of time in the temperature range of 170-300°C; small changes in short-range order cause large changes in the

electrical resistivity; the rate of change of short-range order obeys essentially first-order kinetics; measurements have shown that short-range order is not noticeably destroyed by a few hours of heavy particle radiation. Three deleterious features are the large thermal coefficient of electrical resistivity, the very similar X-ray scattering of copper and zinc, and a tendency to dezincify at high temperatures. The first requires the measurement of resistivity at liquid nitrogen temperature, the second prevents the direct determination of the degree of short-range order, and the third makes annealing at high temperature risky. However, the good features outweigh the bad and this alloy has been used extensively for radiation studies. Details of some of the experiments will now be given.

The black circles of Fig. 1 illustrate the relative equilibrium resistivities of α -brass (30% Zn) measured at liquid nitrogen temperature. Typical times required to achieve these equilibria are indicated. A study of the times required to achieve equilibrium at several temperatures showed that these characteristic times obey an Arrhenius equation (8) and allow the activation energy for the diffusion process to be determined. The energy obtained in this manner is in excellent agreement with that obtained by other techniques. Knowing this, the time to achieve equilibrium at any temperature can be calculated and, as shown in Fig. 1, at 50°C this time would be 30,000 years. When the brass is equilibrated at one of the higher temperatures and then placed in a radiation field at a lower temperature, the enhanced diffusion effect will allow the alloy to achieve equilibrium much faster than under thermal conditions alone, provided the

temperature is still high enough to allow migration of the vacancies. This is illustrated in Fig. 2. The brass was brought to equilibrium at 210°C and then irradiated at 50°C in the reactor and the resistivity decrease measured at intervals in liquid nitrogen (9). This was also done on other samples in a 2 mev electron beam and at 20°C in a 850,000 R/hr ⁶⁰Co source. The final value achieved by six hours of electron radiation shown in Fig. 2 is plotted as the open circle at 50°C in Fig. 1. This clearly shows that very large enhancement of diffusion occurs and that a technique is now available for putting alloys into thermodynamic equilibrium at low temperatures.

The phenomenon of enhanced diffusion has been treated mathematically with some success (7), (10). The diffusion coefficient, D , is given as $D = V_t v_v \lambda^2$ where V_t is the total number of vacancies present, v_v their jump frequency, and λ the jump distance. The number of vacancies present in a radiation field is the sum of V_0 , the thermal vacancies, and V , the radiation produced vacancies, and the central problem in the determination of the diffusion coefficient is the calculation of the total number of vacancies under varying conditions. Three possibilities were considered.

1. The vacancies anneal to internal and external surfaces. The corresponding differential equation is

$$dV/dt = K - K_v V \quad (1)$$

where K is the rate of vacancy production and is proportional to the radiation flux, and K_v is the constant of vacancy escape and is propor-

tional to the vacancy sink concentration, α , and, therefore, $K_V = \alpha v_V \lambda^2$.

2. The vacancies may encounter the interstitials and be annihilated before reaching a sink. The corresponding differential equation is

$$dV/dt = K - V i v_i \quad (2)$$

where i is the concentration of interstitials and v_i is the jump frequency of the interstitials, used here because the interstitial is believed to migrate faster than the vacancy and is, therefore, the rate controlling factor. 3. The vacancies may be removed by both of these processes at the same time, then

$$dV/dt = K - K_V V - V i v_i \quad (3)$$

When the alloy is placed in a radiation field there is a transient condition until a new steady state concentration of vacancies is established. Then, at steady state $dV/dt = 0$ for each of the above equations. The solution to the first, Eq. 1, is $V = K/K_V = K/\alpha v_V \lambda^2$. The diffusion coefficient in the radiation field, D' , contains terms for both the thermal concentration of vacancies, V_0 , and the irradiation produced vacancies, V . Thus

$$D' = (V_0 + V) v_V \lambda^2 = V_0 v_V \lambda^2 + \frac{K v_V \lambda^2}{\alpha v_V \lambda^2} = D + \frac{K}{\alpha} \quad (4)$$

where D is the diffusion coefficient under thermal conditions alone. The natural measure of diffusion enhancement, $D' - D$, is seen to be the tem-

perature independent constant K/α . The solution to this and the other two conditions appear in Table I.

Table I

Activation Energy and Flux Dependence of Radiation

Enhanced Diffusion as Determined by the Annealing Mechanism

<u>Annealing Mechanism</u>	<u>Activation Energy for Enhanced Diffusion</u>	<u>Flux Dependence of Enhanced Diffusion</u>
Linear	0	Linear
Recombination	$E_m^V - (1/2)E_m^I$	Square Root
Recombination plus linear	$(1/2)E_m^V$	Square Root

In this table E_m^v and E_m^i are the migration energies of the vacancies and interstitials respectively. A plot of Eq. 4 is shown by the dashed curve of Fig. 3; the solid line is the thermal diffusion. The ordinate of this figure is $1/\tau$ where τ is the mean atomic jump time, and is related to the diffusion coefficient in f.c.c. metals by

$$D = \lambda^2/12 (1/\tau) \quad (5)$$

Data similar to those of Fig. 2 were taken in the Brookhaven reactor at a variety of temperatures. The reciprocal of the characteristic decay time τ , of the curves, where τ is also the mean atomic jump time, is plotted in Fig. 3 with open circles. It is seen that the behavior predicted by Eq. 4 for the linear annealing of vacancies to sinks is obeyed and, by use of Eq. 5, $D^* = D \approx D^* = K/\alpha \approx 10^{-20} \text{ cm}^2/\text{sec}$.

The validity of this simple theory has been further demonstrated by Dietz and Baltesen in the iron-silicon system (11). They quenched iron containing 3% silicon from 1000°C, where it is soluble, and irradiated it in a reactor at a series of lower temperatures. They studied the precipitation rate by measuring the magnetic aftereffect. Their results are illustrated in Fig. 4 where the plotted variable $n_p D$ is proportional to the $1/\tau$ used in Fig. 3. It is seen that there is a plateau similar to that observed in α -brass between 300 and 400°C. The rapidly decreasing curve below 300°C probably arises from the decreasing mobility of the vacancies where the steady state approximations to Eq. 1-3 are not valid. The slight slope in the plateau region is attributed by the authors to a small contribution from recombination of vacancies and interstitials as shown in Eq. 2 and Table I. From the value of their plateau region, $K/\alpha \approx 10^{-20}$, which is in agreement with the α -brass results. The difference in values between the two 4 cm distance specimens of Fig. 4 probably is caused by a differing α , dislocation concentration, in them. The agreement of their results with the theory also indicates that very little disturbance was caused by the breakup of precipitate clusters.

Arndt and Hines (12) have succeeded in obtaining the case governed by Eq. 3 in which both the linear annealing and the recombination mechanisms are large. They performed experiments on α -brass (10% Zn) subjected to 30 kev He^+ bombardment. They employed the dezincification phenomenon exhibited by α -brass by relating the zinc lost at the surface to the diffusion coefficient. Thus, by observing the comparative rate of change of the optical reflection coefficient of the surface for irradiated and unirradiated samples they were able to measure the enhancement of diffusion by radiation at a series of temperatures. They found that the temperature dependence was not zero as in the linear annealing case, but that the resulting curve could be fitted by the solution of Eq. 3.

As seen in Table 1, the slope for this case is $(1/2)E_m^v$. By this relation their data give $18 \pm 2 \text{ Kcal/mol}$ for the activation energy of vacancy motion. They further verified that the enhanced diffusion was proportional to the square root of the bombarding flux as required in the solution of Eq. 3.

The bombardment of the surface of a metal with neon ions produced 4.5×10^{-2} atomic fractions of defects/sec compared to about 10^{-10} atomic fractions/sec produced in a reactor (?). Such a large concentration of vacancies and interstitials might be expected to permit considerable recombination. It is presumed, therefore, that a light bombardment would produce too low a concentration for recombination and the loss of vacancies would be caused primarily by linear annealing to sinks. Linear annealing should also be favored when the concentration of sinks is increased. If the sink concentration is too high, then the mean free path of the vacancy

is too small to cause much atomic interchange and little or no enhanced diffusion is observed. Arndt and Hines reported that their samples had to be well annealed, and Damask (8) showed that enhanced diffusion in a reactor could not be observed in cold worked brass. Studies of case 1 and case 3 of enhanced diffusion are being made at Brookhaven by the irradiation of α -brass (30% Zn) with ^{60}Co γ -rays. Preliminary results are shown in Fig. 5 where the diffusion is plotted against the γ -ray intensity (13). It is seen that the data can be resolved into two straight lines, one yielding nearly a linear flux dependence and the other a square root flux dependence, with a transition region between. This γ -ray work is not complete nor is it fully interpreted, but we believe that this illustrates the transition of the diffusion enhancement governed by linear annealing, Eq. 1, to that governed by linear annealing plus recombination, Eq. 3. Small amounts of cold work introduced into these specimens drastically reduce the diffusion coefficient, and it is expected that controlled amounts of cold work will cause the transition region to shift to higher fluxes in agreement with the preceding arguments. Because of the difficulty of annealing α -brass the samples under study probably have a high dislocation content and, therefore, the linear annealing case is expected to dominate even at relatively high bombarding fluxes. Other alloys, which can be better annealed, would, therefore, be expected to exhibit some vacancy-interstitial recombination effects at lower fluxes. The work of Wechsler et al on Cu-Al, presented in this session, possibly illustrates this.

Because of the extremely small contribution to the diffusion coefficient from irradiation, all of the above studies involved indirect

measurements of microdiffusion in alloys. One example of the direct observation of enhanced diffusion does exist. The technique was first established by Barnes et al who bombarded copper with α -particles from a cyclotron (14). Subsequent heating produced void formation in the copper within the region of the deposited helium atoms. Their explanation was that the helium precipitates as small gas bubbles in those parts of the metal where the helium atoms receive vacancies from nearby sources. An identical experiment was performed by Coland (15) in which two samples received α -bombardment and then one was heated in the Brookhaven reactor for 10 days at 425°C and the other kept at the same temperature outside. The reactor specimen clearly showed a greater void density which, he concluded, arises from the excess vacancies produced by the neutron irradiation.

C. Radiation Enhanced Nucleation

It was important to the study of radiation enhanced diffusion to isolate it from the other effects of radiation. Because of the successful progress of the understanding of this phenomenon, we began to consider the isolation of radiation enhanced nucleation in alloys. For this study we selected the iron-carbon system. Carbon is known to diffuse interstitially in iron and, therefore, its diffusion rate should not be accelerated (to a first approximation) by the presence of either vacancies or interstitials. Careful studies of thermal precipitation in this system exist (16), and it was hoped that a simple comparison of the relative rates of precipitation in irradiated and unirradiated specimens would yield the increased number of nuclei. The real situation proved to be somewhat more complex.

The rate of disappearance of carbon which had been quenched into solution in iron (0.01 wt. % carbon) was measured by its contribution to the internal friction (Snoek) peak (17). The internal friction peak height was measured at 57°C, by selection of the proper frequency, and its decay observed as a function of time. The samples were then placed in the Brookhaven reactor at 57°C for various times and after removal the observation of the decay rate of the internal friction peak was continued. The results of this experiment are shown in Fig. 6. (18). It is seen that the peak decreases in height while the sample is in the reactor (pre-irradiation values shown on the ordinate) and that after removal from the reactor the remaining internal friction decays at the same time regardless of the time spent in the reactor. The total time at 57°C is shown by the solid points of Fig. 7, and it can be seen that reactor irradiation of four hours accelerates precipitation by a factor of 10 and that further irradiation has no effect. Random walk considerations imply that the number of precipitation nuclei has increased by a factor of 10.

Hull and Mogford (19) performed a similar experiment with electron microscopy. They measured the density and size of precipitate particles in 0.004 wt. % carbon in iron after both thermal and irradiation treatment. After several days of aging at 60°C, the precipitate particle density was about $3 \times 10^{13}/\text{cm}^3$. When quenched iron-carbon was irradiated at 60°C in a neutron flux of similar magnitude to the internal friction work, the particle density increased to $2 \times 10^{14}/\text{cm}^3$ after 5 hours irradiation. Subsequent irradiations up to 144 hours showed essentially no change in the number of particles although their size increased. This independent

observation of a factor of 10 increase in the number of precipitation sites is in excellent agreement with the implications of the internal friction studies. The number of primary knock-ons from such irradiation doses is about $2.5 \times 10^{14}/\text{cm}^3$ after 2 hours. If each forms a nucleation site then not 4, but 2 hours should be sufficient time to yield the above results. It is not known however, why the incipient nuclei created by the primary knock-ons of further irradiation do not form sites of precipitate particles. Further studies of this phenomenon need to be made, particularly with irradiations of under 4 hours duration.

When the quenched iron-carbon is irradiated at low temperature (-100°C) an entirely different effect occurs. Measurements of the rate of decay of the internal friction peak after a 12 day low temperature irradiation show that the carbon disappears over a thousand times faster than in unirradiated iron. Fig. 8 illustrates the normalized curves of this decay at different temperatures after a 12 day irradiation. Similar decays for a 4 day irradiation and no irradiation are also shown for comparison. It should be noted that the decay of the 4 day specimen does not go to completion. This faster rate of disappearance is illustrated also in Fig. 7 in which the 50°C curve of Fig. 8 is plotted and its decay rate at 57°C is calculated for comparison. When the curves of Fig. 8 are analyzed it is found that the disappearance of carbon obeys a second order reaction rate and is governed by the migration energy of carbon in iron, 19.8 Kcal/mol. The number of jumps an average carbon atom makes before disappearance is calculated to be about 20,000. This number requires a far greater density of nuclei than could have been created by the 12 day

irradiation. It is believed, therefore, that the carbon is trapped by point defects, presumably the vacancy, and, although it has not precipitated into a cluster, its contribution to the internal friction peak has been removed. Such a mechanism would explain the number of jumps, the order of reaction, and the energy that controls the reaction. It also indicates that there are insufficient vacancies after a 4 day irradiation to trap all of the carbon atoms in solution in those specimens and, therefore, this decay curve does not show the removal of all of the carbon although it obeys second order kinetics for unequal initial concentration. Irradiations of 19 days have been made and the internal friction peak decay rate is faster than after 12 day irradiations but also obeys second order kinetics for unequal initial concentration. Such a mechanism of carbon trapping would predict that low temperature irradiation would inhibit the formation of precipitates at 40-60°C, not because there are less nucleation sites created, but because the carbon is temporarily trapped and cannot migrate to the sites. Preliminary electron microscope studies of samples irradiated for 19 days at low temperature and annealed for 100 min. at 43°C reveal no trace of precipitation. This observation agrees with the prediction of the interpretation of the low temperature irradiation data given above.

The behavior of the carbon in iron after low temperature irradiation is also being studied by electrical resistivity and a somewhat more revealing picture is emerging (20). Fig. 9 shows the comparison of isochronal annealing of irradiated and unirradiated quenched iron-carbon. The unirradiated specimen shows two distinct steps, one around 170°C and the other around 260°C. The lower temperature step is the one observable by

internal friction techniques and is related to the precipitation of carbon from solution. The high temperature step is associated with the rearrangement of the precipitate into the Fe_3C phase. The irradiated specimen shows considerably more complexity. A 19 day low temperature irradiation has increased the electrical resistivity by about 20% (all resistivities were measured at liquid nitrogen temperature). A low temperature decay occurs around -40°C with an activation energy of about 5 Kcal/mol. This probably arises from the annealing of one of the point defects, possibly the interstitial. A second step occurs around 50°C which corresponds to the internal friction observation. The activation energy of this decay step is 19.8 Kcal/mol and the kinetics are of second order for a slightly unequal initial concentration, in agreement with the internal friction findings. Following this step is a partial step which has not as yet been completely resolved, although its activation energy is around 14 ± 2 Kcal/mol. This step may be related to the precipitation of untrapped carbon and would correspond to the first step of the unirradiated sample which also has about this activation energy. It is hoped that shorter irradiation times will cause an increase in the amount of untrapped carbon and allow this step to be resolved. The higher temperature step is no longer the same as that of the unirradiated case, but now appears to consist of two steps. The first part corresponds in temperature to the high temperature step in that its maximum rate occurs precisely at 260°C and its magnitude is also near that of the unirradiated step. The decay occurring around 310 appears to be a new step produced by the irradiation and unrelated to the effects observed in the unirradiated specimen. It is

perhaps the annealing of residual damage created by neutron irradiation. It is therefore tentatively assumed that at some region the trapped carbon in the irradiated sample boils out of its traps and proceeds to precipitate into Fe_3C in a nearly normal manner. Stored energy measurements currently in progress at Brookhaven may reveal a region of energy absorption that could be assigned to the carbon leaving the traps.

The iron-carbon system had been chosen because the nucleation enhancement phenomenon was expected to be relatively simple, and yet, as was shown, the problem became quite complicated. Further complications may be expected during irradiation at temperatures in between the two described here because both trapping and nucleation should be operative simultaneously. There are several different mechanisms for nucleation and precipitation in alloys, many of which involve vacancy diffusion. The work reported here is, therefore, only a beginning, and studies on many diverse systems must be made before we can both understand the mechanism and obtain quantitative estimates of the enhancement of nucleation by irradiation.

FIGURE CAPTIONS

1. Equilibrium resistivities of α -brass measured at liquid nitrogen temperature. Arrows are example times to achieve equilibrium. Open circle at 50°C is value obtained by electron irradiation. (reference 9).
2. Change in electrical resistivity of α -brass with neutron, electron, and gamma irradiation (reference 9). Fluxes: neutron = 10^{12} nv (fast), electron = 2.6×10^{14} electrons/cm² sec, ⁶⁰Co gamma = 850,000 R/hr.
3. Rate of ordering ($1/\tau$), vs. $1/T$ for neutron irradiation of α -brass. Solid line - thermal rate. Dashed line - theoretical radiation enhanced rate for linear annealing of vacancies. Circles - experimental points (reference 7).
4. Rate of diffusion ($n_0 D$) vs. $1/T$ for reactor irradiation of iron-silicon (reference 11).
5. Rate of ordering, ($1/\tau$), vs. flux for gamma irradiation of α -brass.
6. Decay of internal friction peaks at 57°C after reactor irradiation at 57°C. Pre-irradiation values shown on ordinate. (reference 18).
7. Summary of internal friction data. Solid line labelled Thermal is a composite of peak decay at 57°C of this experiment coupled with the curve at 62°C of Doremus. Dashed line with data points is a curve

through the solid points of total time at 57°C. Open points are irradiation time only, shown for comparison. Curves on left are results of low temperature irradiation. (reference 18).

8. Normalized decay curves of internal friction after low temperature reactor irradiation. (reference 18).
9. Isochronal decay of resistivity of iron carbon quenched from 700°C. (reference 20).

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FIG. 1

FIG. 2

FIG. 3

Please refer to:
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FIG. 4

FIG. 5

FIG. 6

FIG. 7

FIG. 8

FIG. 9