

The Pennsylvania State University
The Graduate School
College of Engineering

DYNAMICS OF FISH

A Thesis in
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by
Your Name

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Abstract

Some nonsense goes here.

Table of Contents

List of Figures	vi
List of Tables	vii
List of Symbols	viii
Acknowledgments	x
Chapter 1	
Ferroelectricity	1
1.1 This is a Section	1
1.1.1 A Table	1
1.1.2 This is a Subsection	1
1.1.3 Some Equations	1
1.2 More Declaration	1
Chapter 2	
Sample Fabrication	2
2.1 Introduction	2
2.2 Flake generation	2
2.2.1 Alignment marker wafer	2
2.2.2 Mechanical flake exfoliation	3
2.3 Photolithography	4
2.3.1 LOR+SPR stack	4
2.3.2 PMMA+PMGI+SPR stack	5
2.4 Thin filament shadow mask	6
2.5 Metal evaporation and liftoff	7
2.6 Wiring	7
2.7 Layer Transfer?	8
Appendix A	
Title of the First Appendix	10
A.1 Introduction	10
A.2 More Declaration	10

A.2.1	Some Subsection Title Here	10
Appendix B		
	Title of the Second Appendix	12
B.1	Introduction	12
B.2	More Declaration	12
Appendix C		
	Title of the Third Appendix	14
C.1	Introduction	14
C.2	More Declaration	14
Appendix D		
	Title of the Fourth Appendix	16
D.1	Introduction	16
D.2	More Declaration	16
Appendix E		
	Title of the Fifth Appendix	18
E.1	Introduction	18

List of Figures

2.1	TODO: Device manufacture.	2
-----	-----------------------------------	---

List of Tables

2.1	TODO: Lithography resists	5
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List of Symbols

- α The first greek letter, p. ??
- α The first greek letter, p. ??
- α The first greek letter, but we should really add some more text, though we need it to go on two lines, p. ??
- α The first greek letter, p. ??
- α The first greek letter, p. ??
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Acknowledgments

Chapter 1 | Ferroelectricity

1.1 This is a Section

1.1.1 A Table

1.1.2 This is a Subsection

1.1.3 Some Equations

1.2 More Declaration

Chapter 2 |

Sample Fabrication

2.1 Introduction

TODO: Explain about the 2D fad. Overall device construction.

2.2 Flake generation

2.2.1 Alignment marker wafer

Before fabricating any devices to test materials we needed a platform to work on that would make it easy to find flakes and manufacture devices. For this 4 inch wafers of $500\mu m$ phosphorus doped silicon with $300nm$ of thermally oxidized SiO_2 was chosen as a substrate (purchased from Si-Tech, $\langle 100 \rangle$ face, $\rho \lesssim 5m\omega \cdot cm$). The high conductivity and thermal oxide allowed us to construct a backgated field effect transistor (see: FIXME). The substrate was polished for better adhesion of deposited flakes. Using standard photolithography (see 2.3.1) we patterned the surface with a $\sim 5nm$ thin layer of titanium for adhesion then $\sim 35nm$ of gold. The metals were laid out with a repeating pattern of numbers and lines to easily identify the location of a flake on the substrate when moving between tools. We spun a layer of PMMA resist over the substrate then

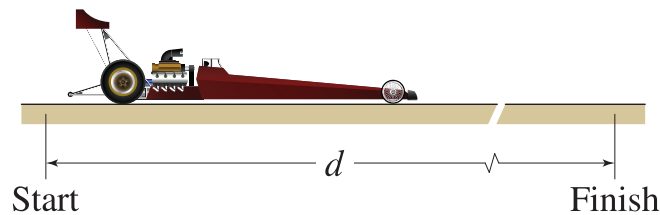


Figure 2.1. TODO: Device manufacture.

diced the wafer $8mm$ square chips with a ADT 7100 ProVectus Dicing Saw. This allowed us to make many devices per wafer while customizing their orientation and pattern for each flake. The resist was then removed from the chip using acetone, 2-isopropanol, and deionized (DI) water then dried using a nitrogen blow gun. To remove any residue organics we soaked the chips in Nano-Strip (sulfuric acid and hydrogen peroxide compound, VWR) for 10 minutes, rinsed in DI-water, then repeated the initial solvent clean. After several devices experienced dielectric breakdown across the SiO_2 we changed the residue clean to an oxygen surface treatment. This was done in a M4L Plasma Processing System with 300 sccm oxygen, 100 sccm helium, at 500mTorr of pressure, and 100W for 1 minute.

2.2.2 Mechanical flake exfoliation

Flakes were exfoliated using Scotch 3M Transparent Tape. The tape was pressed into a crystal of the desired material then peeled off, resulting in a few smaller chunks of crystals adhered to the tape. The tape was then folded over itself so the crystals were pressed into two different areas of tape. The tape was then peeled apart so that the crystals would be pulled apart into multiple chunks. This process was repeated several times to provide a region roughly 8 to $10mm$ in diameter with crystal coverage. This region was then pressed into a fresh region of tape so the exposed surface of crystals was free of tape residue. The tape and crystals was then placed on a cleaned alignment marker chip to maximize the amount of crystals within the center of the chip. The tape was lightly pressed into the chip so that the crystals would be in contact with the surface of the chip. To promote adhesion to the surface, the chip was then heated for 2 minutes at $100C$. [1] The chips were then allowed to cool and the tape peeled off.

After exfoliation we scanned each chip for flakes using a Nikon Eclipse LV150 microscope. We chose $300nm$ of thermally oxidized SiO_2 to increase visibility of 2D materials under white light. [2] Flakes choice was optimized for uniformity, thinness, and lateral sizes large enough for photolithography processing. A Bruker Icon Atomic Force Microscope was used to measure flake height and develop a thickness color-code to compare new exfoliated flakes against.

TODO: tape figure, alignment mask?

2.3 Photolithography

Photolithography is a common technique used by industry and academia alike. In most variants of photolithography, a series of photosensitive polymers, or resists, are spread over a surface then exposing the polymer to light to change the chemical makeup of the polymer. This can be done by spinning a solution of the desired polymer, dissolved within a compatible solvent, at high speeds over a substrate. The solution is dripped on the substrate either statically or dynamically, under a slower rotation. The substrate is then rotated at high speeds until the desired thickness of polymer is left on the surface, the excess being flung off the substrate edges. The solvent can then be removed by baking the substrate and polymer for a short time until a desired durability is achieved. After exposure the resist is then removed by soaking the substrate in a chemical developer. Developer selection must take into account any chemistry between the developer and flake material as some developers can react with the exposed material. For negative resists, light exposed regions of polymer are removed when developed, positive resists will only have non-exposed regions removed. For devices that change based on the flake, the resist pattern is generated on a computer and then written by a moving laser to fit the device dimensions. In commonly used patterns, a mask of chrome on quartz is prefabricated. The mask is moved over the flake then the entire surface is flooded with light at once, exposing the regions with the desired resist pattern. We typically choose a multiple layer resist stack as allowing the bottom layer dissolve long enough to undercut the top allows for easier liftoff when removing evaporated metals (See 2.5). Resist can also be used to protect part of a sample when etching allowing a patterned etch of the substrate or testing device.

2.3.1 LOR+SPR stack

The LOR+SPR negative resist stack is easy to use and allows for high resolution patterns down to $2\mu m$. Before any resist is added the substrate is given a dehydration bake of $115^{\circ}C$ for 60s to remove any water. A layer of LOR-2A is statically deposited as the first layer of the stack. The substrate is then spun at $4000RPM$ for 45s reducing the LOR-2A to a $\sim 200nm$ thick coating. [3] Finally we bake it at $180^{\circ}C$ for 60s on a hot plate to remove some solvent. Next a layer of SPR-3012 is dynamically dispensed at 900RPM. The substrate is then sped up and spun again at $4000RPM$ for 45s flattening the SPR-3012 to $\sim 1.3\mu m$ thick. [4] This is then baked for $95^{\circ}C$ for 60s on a hot plate. The resist stack and flake is then exposed using a MA/BA Gen4 or Gen2 Contact Aligner

(SUSS MicroTec) with 54mW of light. The patterned resist is then developed for 60s in Microposit CD-26 (Tetramethylammonium hydroxide 1-5% and water, Shipley).

TODO resist stack

Table 2.1. TODO: Lithography resists

Resist	Solvent	Developer	Liftoff
LOR-2A		CD-26	Remover-PG
SPR-3012		CD-26	Remover-PG
PMMA		MIBK	Acetone/Remover-PG
SF6-slow		101A	Remover-PG

2.3.2 PMMA+PMGI+SPR stack

We found some chalcogenides reacted to alkali developers such as CD-26 [5] so we developed an alternate photolithography resist stack with a solvent used to develop the material layer. First a layer of Polymethyl methacrylate (PMMA) (-2A Microchem) is statically deposited, spun at 2500*RPM* for 45s to give a $\sim 600nm$ layer, then baked at 180C for 180s. [6] Next a layer of PMGI SF6-slow is dynamically spun at 900*RPM*, then 4000*RPM* for 45s to reduce the thickness to $\sim 300nm$, and baked at 180C for 60s. [7] Finally, a top-coat of SPR-3012 is dynamically dispensed at 900*RPM*. This is then spun at 4000*RPM* for 45s for a thickness $\sim 1.3\mu m$ and baked for 95C for 60s. The SPR-3012 is then exposed using a MA/BA Contact aligner with 54mW of light (peak 365nm), developed for 60s in in Microposit CD-26 from Shipley (2.3.1), then rinsed in water. The PMMA and SF6 under-layers are exposed for 30 minutes in a Deep UV (peak 270nm, FIXME WHO) at 5.4W (FIXME wavelength and energy). The SF6 is developed in 101A developer (Microchem) for 90s then rinsed in water. Finally the PMMA is developed for 10 minutes in methyl isobutyl ketone (MIBK) and a trace amount of water. The substrate is moved though the water for 30 – 60s to remove a thin film that forms, causing the film to pull off. The MIBK is then removed with 2-proponal.

An alternate version of this process used a thicker PMMA (6A) with SPR-3012 on top. Because the SPR uses a PMMA-compatible solvent (Cyclopentanone) the two layers would intermix and form a layer that was unable to be dissolved in either developer. This was removed by ashing the layer for 8:35 minutes in the M4L with 250 sccm oxygen, 50 sccm helium, 500mTorr pressure, 450W power. This process varied greatly depending

on polymer uniformity and location of device on the substrate so later devices included the PMGI SF6 layer to eliminate the inconsistency.

TODO: figure resist stack

2.4 Thin filament shadow mask

As an alternative to lithography, we used an all-dry quartz filament shadow mask for metal evaporation (2.5). This allowed us to test contact resistance of an underlying material without introducing the polymers, chemicals, and processing to the material that may damage the surface or leave a residue between the material and metal contacts. We heated two quartz rod ends with an acetylene torch until semi-molten. We then lightly touched the two ends together and quickly pulled the ends apart within the flame. If done correctly, this produced several 2 – 10cm long strands of quartz that were $< 2\mu m$ in diameter. These were identified under a florescent light by their thread-like behavior, losing most stiffness and free to float in the air when $\lesssim 2\mu m$ in diameter. These filaments were then caught on glass forks by dragging the filament across the opening and snapping the end attached to the quartz rod. The forks were cut to fit over the sides of our sample chip substrates and reinforced with metal for durability. The sample chip was attached to a glass slide with double-sided tape. These filaments were then draped over the sample material with the glass fork. The ends of the filament were then cut with a tungsten needle and the glass fork removed. The filament could be adjusted by pulling it gently with a thin copper wire until it was in place and laying flat against the surface. This often caused the quartz to snap so the range of movement was limited to within a few 10s of microns from where it started. Once the filament was maneuvered it was fixed in place by wrapping the ends in Teflon tape that wrapped around the glass slide. Teflon tape was chosen as it is unlikely to scratch the material and has no adhesive that would cause the filament to move when securing it. The tape was also used to reduce the exposed area so only a small region around the material was included and reduce the risk of a short between the two sides. We found a filament less than $.5\mu$ tended to cause a short between the two sides while greater than $3\mu m$ would not sit flush against the surface. Metal choice is similarly important as certain metals like indium tended to “wet” across the surface and short at any diameter.

TODO: figure filament device, undercut with indium

2.5 Metal evaporation and liftoff

Metal deposition was done using the Lab-18 Thin Film Deposition System (Kurt J. Lesker Co.). This was used to add contacts to a lithography-patterned flake by covering a substrate with layers of metal. We used electron beam evaporated titanium and gold for the uniform deposition and high conductivity. First $\sim 5nm$ of titanium was deposited at $\sim 1\text{\AA}/s$ as titanium adheres well to SiO_2 . Next a layer of $35 - 50nm$ of gold was evaporated at $\sim 1\text{\AA}/s$ on top of the titanium. The gold provided a highly conductive protective layer that was inert in atmosphere and safe to use when wiring samples.

The Lab-18 Thin Film Deposition System also had an in situ ion source that could be used to clean and roughen the surface of a material before deposition occurred. A typical process used $10sccm$ of argon gas flowed over a metal coil with $2.5A$ of emission current and $150V$ between the coil and the substrate. The ion source is undirected which caused trace amounts of iron from the chamber itself to be knocked loose and deposited on the surface of the sample.

To remove excess metals from the substrate the lithography resist was then removed through a 'liftoff' process. The resist stacks were designed to have a small undercut between the top and bottom layer to allow solvents to dissolve the resists from the edges even if metal had deposited on the sides of the upper layers. For LOR-based (see 2.3.1) stacks the substrate was soaked in heated Remover-PG (Microchem) for 10 minutes at $60C$. The Remover-PG was then gently blown with a pipette over the surface of the substrate to push away any metals that were no longer adhered to resist. This process was repeated until all of the resist and metal adhered to it were removed. As smaller features had less surface area of resist for the solvent to resolve, more forceful streams were sometimes needed. If this was not enough to peel off the resist, sonication in a water bath was sometimes used. This often pulled off the metals deposited on the substrate, flake, or even the flake itself, so was used only when necessary. The Remover-PG was then removed by soaking the material in 2-propanol for 5 minutes and blow-dried with a nitrogen gun. For PMMA based stacked unheated acetone was used in the place of Remover-PG while the rest of the process was the same.

2.6 Wiring

Lithography wiring patterns were designed with $.5 - 1mm$ square pads that connected to metal leads on the sample material. To connect this to a preexisting wiring system

we used a series of indium dots and thin gold wires (99.95% Au, $25\mu m$, Alfa Aesar). To avoid any currents damaging the sample, we first adhered the wire to the wiring structure with lead-free solder. Next we placed an indium dot (FIXME source) on each of the sample wiring pads using a modified tweezer. The tweezer was cut so that only one prong remained, then the tip cut flat and further sanded smooth with the side of a razor blade. This allowed the indium dot to adhere to the tip easily and for the modified tweezer to be held similar to a pen for increased dexterity and use with samples in recessed mounts. The gold wire was then bent down and pushed into the indium dot and a second indium dot was pressed into the first, forming a sandwich-like structure, that held the gold wire in place. The gold wire was then bent from the sample to prevent it from coming into contact with any part of the sample or other wires. This process was repeated for each of the wiring pads on the device.

To prevent static discharge from damaging the flake several measures were taken. A ionizing fan (Chapman VSE3000 static eliminator) was constantly blown across the sample and wiring station at all times. The person wiring was grounded with a static wrist strap to the building ground at all times when touching the device. The modified tweezer was held without gloves to connect it to the building ground through the body. The wiring station used a rubber floor mat with metal stool to isolate the person from the floor and prevent any charge buildup from the ground. Finally, clothing was chosen to reduce the buildup of static, especially in winter as the dryer air made hazardous amounts of static buildup fairly frequent.

TODO: figure, wiring

2.7 Layer Transfer?

Appendix A |

Title of the First Appendix

A.1 Introduction

When in the Course of human events, it becomes necessary for one people to dissolve the political bands which have connected them with another, and to assume among the powers of the earth, the separate and equal station to which the Laws of Nature and of Nature's God entitle them, a decent respect to the opinions of mankind requires that they should declare the causes which impel them to the separation.

A.2 More Declaration

We hold these truths to be self-evident, that all men are created equal, that they are endowed by their Creator with certain unalienable Rights, that among these are Life, Liberty and the pursuit of Happiness. –That to secure these rights, Governments are instituted among Men, deriving their just powers from the consent of the governed, –That whenever any Form of Government becomes destructive of these ends, it is the Right of the People to alter or to abolish it, and to institute new Government, laying its foundation on such principles and organizing its powers in such form, as to them shall seem most likely to effect their Safety and Happiness.

A.2.1 Some Subsection Title Here

Prudence, indeed, will dictate that Governments long established should not be changed for light and transient causes; and accordingly all experience hath shewn, that mankind are more disposed to suffer, while evils are sufferable, than to right themselves by abolishing the forms to which they are accustomed. But when a long train of abuses and usurpations, pursuing

invariably the same Object evinces a design to reduce them under absolute Despotism, it is their right, it is their duty, to throw off such Government, and to provide new Guards for their future security. –Such has been the patient sufferance of these Colonies; and such is now the necessity which constrains them to alter their former Systems of Government. The history of the present King of Great Britain [George III] is a history of repeated injuries and usurpations, all having in direct object the establishment of an absolute Tyranny over these States. To prove this, let Facts be submitted to a candid world.

Appendix B |

Title of the Second Appendix

B.1 Introduction

When in the Course of human events, it becomes necessary for one people to dissolve the political bands which have connected them with another, and to assume among the powers of the earth, the separate and equal station to which the Laws of Nature and of Nature's God entitle them, a decent respect to the opinions of mankind requires that they should declare the causes which impel them to the separation.

B.2 More Declaration

We hold these truths to be self-evident, that all men are created equal, that they are endowed by their Creator with certain unalienable Rights, that among these are Life, Liberty and the pursuit of Happiness. –That to secure these rights, Governments are instituted among Men, deriving their just powers from the consent of the governed, –That whenever any Form of Government becomes destructive of these ends, it is the Right of the People to alter or to abolish it, and to institute new Government, laying its foundation on such principles and organizing its powers in such form, as to them shall seem most likely to effect their Safety and Happiness. Prudence, indeed, will dictate that Governments long established should not be changed for light and transient causes; and accordingly all experience hath shewn, that mankind are more disposed to suffer, while evils are sufferable, than to right themselves by abolishing the forms to which they are accustomed. But when a long train of abuses and usurpations, pursuing invariably the same Object evinces a design to reduce them under absolute Despotism, it is their right, it is their duty, to throw off such Government, and to provide new Guards for their future security. –Such has been the patient sufferance of these

Colonies; and such is now the necessity which constrains them to alter their former Systems of Government. The history of the present King of Great Britain [George III] is a history of repeated injuries and usurpations, all having in direct object the establishment of an absolute Tyranny over these States. To prove this, let Facts be submitted to a candid world.

Appendix C |

Title of the Third Appendix

C.1 Introduction

When in the Course of human events, it becomes necessary for one people to dissolve the political bands which have connected them with another, and to assume among the powers of the earth, the separate and equal station to which the Laws of Nature and of Nature's God entitle them, a decent respect to the opinions of mankind requires that they should declare the causes which impel them to the separation.

C.2 More Declaration

We hold these truths to be self-evident, that all men are created equal, that they are endowed by their Creator with certain unalienable Rights, that among these are Life, Liberty and the pursuit of Happiness. –That to secure these rights, Governments are instituted among Men, deriving their just powers from the consent of the governed, –That whenever any Form of Government becomes destructive of these ends, it is the Right of the People to alter or to abolish it, and to institute new Government, laying its foundation on such principles and organizing its powers in such form, as to them shall seem most likely to effect their Safety and Happiness. Prudence, indeed, will dictate that Governments long established should not be changed for light and transient causes; and accordingly all experience hath shewn, that mankind are more disposed to suffer, while evils are sufferable, than to right themselves by abolishing the forms to which they are accustomed. But when a long train of abuses and usurpations, pursuing invariably the same Object evinces a design to reduce them under absolute Despotism, it is their right, it is their duty, to throw off such Government, and to provide new Guards for their future security. –Such has been the patient sufferance of these

Colonies; and such is now the necessity which constrains them to alter their former Systems of Government. The history of the present King of Great Britain [George III] is a history of repeated injuries and usurpations, all having in direct object the establishment of an absolute Tyranny over these States. To prove this, let Facts be submitted to a candid world.

Appendix D |

Title of the Fourth Appendix

D.1 Introduction

When in the Course of human events, it becomes necessary for one people to dissolve the political bands which have connected them with another, and to assume among the powers of the earth, the separate and equal station to which the Laws of Nature and of Nature's God entitle them, a decent respect to the opinions of mankind requires that they should declare the causes which impel them to the separation.

D.2 More Declaration

We hold these truths to be self-evident, that all men are created equal, that they are endowed by their Creator with certain unalienable Rights, that among these are Life, Liberty and the pursuit of Happiness. –That to secure these rights, Governments are instituted among Men, deriving their just powers from the consent of the governed, –That whenever any Form of Government becomes destructive of these ends, it is the Right of the People to alter or to abolish it, and to institute new Government, laying its foundation on such principles and organizing its powers in such form, as to them shall seem most likely to effect their Safety and Happiness. Prudence, indeed, will dictate that Governments long established should not be changed for light and transient causes; and accordingly all experience hath shewn, that mankind are more disposed to suffer, while evils are sufferable, than to right themselves by abolishing the forms to which they are accustomed. But when a long train of abuses and usurpations, pursuing invariably the same Object evinces a design to reduce them under absolute Despotism, it is their right, it is their duty, to throw off such Government, and to provide new Guards for their future security. –Such has been the patient sufferance of these

Colonies; and such is now the necessity which constrains them to alter their former Systems of Government. The history of the present King of Great Britain [George III] is a history of repeated injuries and usurpations, all having in direct object the establishment of an absolute Tyranny over these States. To prove this, let Facts be submitted to a candid world.

Appendix E |

Title of the Fifth Appendix

E.1 Introduction

When in the Course of human events, it becomes necessary for one people to dissolve the political bands which have connected them with another, and to assume among the powers of the earth, the separate and equal station to which the Laws of Nature and of Nature's God entitle them, a decent respect to the opinions of mankind requires that they should declare the causes which impel them to the separation.

Some text.

program chaos

c When a LS Fortran **program** has been compiled **and** linked into Mac
c application, **all** information written **to** the screen **WRITE(6,...)** or
c **WRITE(*,...)** appears **in** a standard Mac window, complete with basic
c menus.

external fex, jac

double precision atol, rtol, rwork, t, tout, h

double precision tttotal, dtout

dimension h(3), atol(3), rwork(70), iwork(23)

character*8 tstart, tend

neq = 3

call time(tstart)

write(6,*) "begin_integration_at_", tstart

write(6,*)

c — **Read in** the total initial angular momentum. The total angular
c momentum H is always unity due **to** normalization.

open(**unit** = 2, **file** = 'chaos.data', **status** = 'unknown')

read(2,*) h(1), h(2), h(3)

c — The integration begins at $t = 0$ **and** the values are printed at
c every tout. tout is incremented below. tttotal is the length
c of the entire integration. The **number** of recorded values of
c the integration is given by npoints.

t = 0.0d0

tout = 0.0d0

write(6,*) 'Duration_of_integration_interval, i.e., tfinal?'

read(6,*) tttotal

write(6,*)

write(6,*) 'Number_of_points_for_trajectory_plot?'

read(6,*) npoints

write(6,*)

dtout = tttotal/dfloat(npoints)

```

    tout = tout + dtout

c ——— Tolerance parameters used by lsoda.
    itol = 2
    rtol = 1.0d-9
    atol(1) = 1.0d-9
    atol(2) = 1.0d-9
    atol(3) = 1.0d-9

c ——— Other parameters used by lsoda. See below.
    itask = 1
    istate = 1
    iopt = 1
    lrw = 70
    liw = 23
    jt = 1

    do 11 kount = 5,10
        rwork(kount) = 0.0d0
        iwork(kount) = 0
11  continue
    iwork(6) = 100000

    open(unit = 3, file = 'traj.dat', disp = 'keep',
&      status = 'unknown')

c ——— The actual integration begins here. Loop on the value of iout.
    do 40 iout = 1, npoints

        call lsoda(fex, neq, h, t, tout, itol, rtol, atol, itask, istate,
&                iopt, rwork, lrw, iwork, liw, jdum, jt)

c ——— Write the output to the file traj.dat.
    write(3,20) t, h(1), h(2), h(3)
20  format(f9.1, 3e15.6)

```

```

        if (mod(tout,5000.0d0) .eq. 0.0d0) then
            write(6,*) tout
        end if

c ——— Check to see that things are going OK.
        if (istate .lt. 0) go to 80

c ——— Set the time at which the integration is next recorded and
c      continue the do-loop.
40      tout = tout + dtout

        write(6,*) 'number_of_steps_taken:', iwork(11)
        write(6,*) 'number_of_f_evaluations:', iwork(12)
        write(6,*) 'number_of_Jacobian_evaluations:', iwork(13)
        write(6,*) 'method_order_last_used:', iwork(14)
        write(6,*) 'method_last_used(2==stiff):', iwork(19)
        write(6,*) 'value_of_t_at_last_method_switch:', rwork(15)
        write(6,*)

        call time(tend)
        write(6,*) "end_integration_at", tend
    stop

c — If there is an error, given by istate < 0, write the following.
80  write(6,90) istate
90  format(///22h error halt.. istate =,i3)

    stop
end

```

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Vita

Your Name

The details of my childhood are inconsequential.